

EPA Region 5 Records Ctr.



230156

**Bottom Sediment Sampling and Analysis Report for
the Pumping Lagoon, Industrial Canal, Collection
Basin, and Borrow Pit**

**Johns Manville
1871 North Pershing Road
Waukegan, Illinois 60087**

September 12, 2002

LFR
LEVINE • FRICKE

September 12, 2002

Mr. Brad Bradley
Project Manager
United States Environmental Protection Agency Region 5
77 West Jackson Boulevard
Chicago, IL 60604-3507

Subject: Letter of Transmittal; Bottom Sediment Sampling and Analysis Report for the Pumping Lagoon, Industrial Canal, Collection Basin, and Borrow Pit; September 12, 2002;
Second Explanation of Significant Differences; Johns Manville, Waukegan, Illinois

Dear Mr. Bradley:

Enclosed are 4 copies of the Bottom Sediment Sampling and Analysis Report for the Pumping Lagoon, Industrial Canal, Collection Basin, and Borrow Pit. If you have any questions, please contact me at (847) 695-8855, extension 101.

Sincerely,



William A. Bow C.P.G.
Principal

Attachment

cc: Denny Clinton/JM
Bruce Ray/JM
Ed Kenney/Sidley & Austin
Dave Peterson/DMP PE PC
Elizabeth Wallace/IAG
Sandy Bron/IEPA
Don Gimbel/IEPA
Ann Coyle/U.S. EPA

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the Pumping Lagoon, Industrial Canal, Collection
Basin, and Borrow Pit**

**Johns Manville
1871 North Pershing Road
Waukegan, Illinois 60087**

September 12, 2002

009-07992-00

Prepared for
W. D. Clinton
Johns Manville
1871 North Pershing Road
Waukegan, Illinois 60087



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1.0 INTRODUCTION

On September 22, 2000, the United States Environmental Protection Agency (U.S. EPA) issued a Second Explanation of Significant Differences (2nd ESD) for the Johns Manville (JM) facility in Waukegan, Illinois (the "Site"). Among other requirements, the 2nd ESD stated that "sediments in the Collection Basin, Industrial Canal, and the Pumping Lagoon must be sampled for asbestos." The samples were collected on June 18 and 19, 2002 pursuant to the following work plan and amendment letters:

- *Sediment Sampling and Analysis Plan and Conceptual Closure Schedule*; Letter from Johns Manville, February 14, 2002
- *Response Letter*; Letter from U.S. EPA, April 29, 2002
- *Letter of Transmittal for U.S. EPA Region 1 Standard Operating Procedure for Screening Analysis of Soil and Sediment Samples for Asbestos Content*, Letter from LFR, May 6, 2002
- *Sediment Sampling*, Letter from LFR, June 11, 2002

Copies of the above correspondence may be found in Appendix A.

This report presents the sediment sampling results.

1.1 Site Description

The JM facility is located along the shoreline of Lake Michigan in the City of Waukegan, Lake County, Illinois (see Figure 1, Site Location Map). The Site is bounded on the west by the Union Pacific Railroad, on the south by Midwest Generation and City of Waukegan property, on the east by Lake Michigan, and on the north by Illinois Beach State Park. Figure 2 presents a General Property Map showing the main features of the Site, including the locations of the Industrial Canal, Pumping Lagoon, Collection Basin, and Borrow Pit.

1.2 Water Body Description and Historical Use

1.2.1 Industrial Canal and Pumping Lagoon

The Industrial Canal (approximately 14.9 acres) and Pumping Lagoon (approximately 4.2 acres) were most likely excavated during the 1920s to provide fill materials (sand) during construction of the manufacturing buildings. Although separated by a road, they are hydraulically connected by a submerged culvert located beneath the road, therefore they exhibit the same water elevation at approximately 580 feet above sea level (ASL). The water elevation is supported by surrounding groundwater discharge from the parkland to the north and the JM property to the south, stormwater discharge from the

City of Waukegan, and adjacent Lake Michigan (currently at approximately 578.4 feet ASL).

Maximum water depths along the centerline of each water body are approximately 17 to 19 feet.

Historically, the Industrial Canal and Pumping Lagoon served as part of the plant's "manufacturing water" treatment system. Water was pumped from the western end of the Pumping Lagoon for use in the production of many of the products previously manufactured at the Waukegan plant. Excess "manufacturing water" was pumped to the Settling Basin via the plant's stormwater system. The Settling Basin enabled (via residence time) excessive suspended solids to "settle out" before the water was returned via the Collection Basin to the eastern end of the Industrial Canal. Continuous withdrawals from the Pumping Lagoon induced water flow from the eastern end of the Industrial Canal back towards the west, thereby essentially completing a "manufacturing water" cycle.

1.2.2 Collection Basin

The Collection Basin, currently 5.5 acres in size, was historically part of the "manufacturing water" treatment system at the site. Water exiting the Settling Basin passed through the Collection Basin before being returned to the eastern end of the Industrial Canal. The elevation of water surface in the Collection Basin is approximately 588 to 590 feet ASL, which is supported through seepage from the Settling Basin. The maximum water depth is approximately 5 to 6 feet.

1.2.3 Borrow Pit

The Borrow Pit water body was formed from periodic excavation as a source of sand, most recently during the early 1990s for closure of the CERCLA landfill located on the eastern end of the JM property. The Borrow Pit never served as part of the historic "manufacturing water" treatment system at the site. At present, the water body is approximately 31 acres in size and 16 feet in depth at its deepest point. Similar to the Industrial Canal and Pumping Lagoon, the water elevation is supported at approximately 580 feet ASL by surrounding groundwater discharge, stormwater discharge from the City of Waukegan, and nearby Lake Michigan (currently at approximately 578.4 feet ASL). There is an open culvert that forms a hydraulic connection between the Borrow Pit and the Industrial Canal.

2.0 SEDIMENT SAMPLE COLLECTION

2.1 Sample Locations

Sediment samples were collected from a total of 21 locations in the 4 water bodies. The locations were grouped along 7 separate transects:

- Pumping Lagoon: Transect Line 1; 3 sample locations
- Industrial Canal: Transect Lines 2, 3, and 4: 9 sample locations
- Collection Basin: Transect Lines 5 and 6: 6 sample locations
- Borrow Pit: Transect Line 7: 3 sample locations

Figure 3 depicts each sediment sample location.

As part of the project documentation, water depth and bottom sediment thickness were measured at each sample location. Water depth was measured using a hand-held sonic depth indicator. With the exception of the Collection Basin, sediment thickness was calculated by inserting a 20-foot section of ½-inch steel rebar to “hard bottom,” assumed to be the underlying native sand. By using the water depth and measuring the amount of rebar that extended above the surface of the water, the sludge thickness was calculated. In the Collection Basin, it was not possible to insert the rebar more than a few inches, indicating the presence of sandy or other hard sediments immediately beneath the bottom sediment surface. Therefore, no sediment thickness was reported for the Collection Basin. The water depth and sediment thickness data is presented in Table 1.

2.2 Sample Collection

The samples were collected from a 16-foot open-hull boat equipped with an electric motor. Once the boat was stabilized at a sample station, its position was estimated by observing the distance to each shore.

The samples were collected at each sample station using a stainless steel sediment sampler supplied by AMS, Inc. The 12-inch long by 3 ¼-inch outer diameter core barrel was equipped with an auger tip incorporated with a one-way valve which allows sediment into the sampler, but prevents it from escaping by closing as the sampler is withdrawn. Five-foot long carbon steel extension rods were threaded together and topped with a cross handle so that the core barrel could be lowered through the water and driven into the sediment. Samples were transferred from the core barrel directly into 12 inch long by 3-inch diameter plastic liners and each end was fitted with polyethylene caps. The samples were labeled with the station location and time. The core barrel was decontaminated between sample stations by rinsing in water. Samples

were transferred into clean, labeled 4-ounce glass jars and shipped under chain-of-custody protocols for analysis at EMSL Analytical, Inc. of Westmont, New Jersey. A total of 21 samples plus 1 duplicate were collected.

In all cases, the bottom sediments may be described as a well sorted, fine to very fine grained, black to blackish-gray sludge.

2.3 Laboratory Analysis

The sediment samples were analyzed for asbestos fiber using the U.S. EPA Region 1 Standard Operating Procedure for Screening Analysis of Soil and Sediment Samples for Asbestos Content, as modified by EMSL. This protocol (see Appendix A) consists of a sample preparation step, then analysis of asbestos content using polarized light microscopy (PLM). Results are reported in percent asbestos.

3.0 ANALYTICAL RESULTS

Table 2 presents a summary of the analytical results. The results are depicted on Figure 4. The laboratory report and chain-of-custody forms are presented in Appendix B.

3.1 Pumping Lagoon

Results from the 3 sediment samples collected from the Pumping Lagoon ranged from less than 0.25 percent total asbestos (PL-1C) to 6.8 percent total asbestos (PL-1A). The third sample exhibited a total asbestos content of 4.7 percent (PL-1B). In summary, 2 of the 3 sediment samples collected from the Pumping Lagoon exhibited a total asbestos content greater than 1 percent. Water depths at the sample locations ranged between 12.9 and 13.8 feet.

3.2 Industrial Canal

Results from the 9 sediment samples collected from the Industrial Canal ranged from 1.0 percent total asbestos (IC-4B) to 5.7 percent total asbestos (IC-2C). In summary, all 9 sediment samples collected from the Industrial Canal exhibited a total asbestos content equal to or greater than 1 percent. Water depths at the sample locations ranged between 14.5 and 17.5 feet.

3.3 Collection Basin

Results from the 6 sediment samples collected from the Collection Basin ranged from "none detected" (CB-6B) to 3.0 percent total asbestos (CB-5B). In summary, 4 of the 6 sediment samples collected from the Collection Basin exhibited a total asbestos content equal to or greater than 1 percent. Water depths at the sample locations ranged between 4.5 and 5.1 feet.

3.4 Borrow Pit

Results from the 3 sediment samples collected from the Borrow Pit ranged from "none detected" in 1 sample (BP-7C) to less than 0.25 percent chrysotile asbestos (BP-7A and BP-7B). In summary, none of the sediment samples collected from the Borrow Pit exhibited a total asbestos content equal to or greater than 1 percent. Water depths at the sample locations ranged between 11.3 and 13.2 feet.

4.0 DISCUSSION OF RESULTS

4.1 Pumping Lagoon

The analytical results showed that the bottom sediments in the Pumping Lagoon have been affected by asbestos. This is consistent with the use of this water body as part of the plant's historical "manufacturing water" treatment system. Bottom sediment thickness in the deepest portion of the Pumping Lagoon ranged from 1.2 feet to 4.2 feet. Based upon visual observation, bottom sediments affected by asbestos are expected to thin with the shallower water depths along the shoreline of the Pumping Lagoon.

4.2 Industrial Canal

The analytical results showed that the bottom sediments in the Industrial Canal have been affected by asbestos. This is consistent with the use of this water body as part of the plant's historical "manufacturing water" treatment system. Bottom sediment thickness in the deepest portion of the Industrial Canal ranged from 0.5 feet to greater than 3.3 feet. Based upon visual observation, bottom sediments affected by asbestos are expected to thin with the shallower water depths along the shoreline of the Industrial Canal.

4.3 Collection Basin

The analytical results showed that the bottom sediments in the Collection Basin have been affected by asbestos. This is consistent with the use of this water body as part of the plant's historical "manufacturing water" treatment system. Regarding the thickness of the bottom sediments and/or non-native materials, nearby soil borings place the elevation of the underlying native sand at approximately 580 feet ASL. As the elevation of the bottom sediment in the deepest portion of the Collection Basin is approximately 583 to 584 feet ASL, approximately 3 to 4 feet of non-native sediments/soil are present in the deepest portion of the Collection Basin, with a commensurate increase in thickness with lesser water depths.

4.4 Borrow Pit

The analytical results showed that the bottom sediments in the Borrow Pit do not exhibit asbestos content greater than 0.25 percent. Asbestos would not be expected in the bottom sediments of this water body, as it was not historically used as part of the plant's "manufacturing water" treatment system.

The lack of asbestos fibers within the bottom sediments of the Borrow Pit is further supported by water samples that were collected in April 2000. On April 12, 2000 water samples were collected from 6 locations within the Borrow Pit (see Figure 5). Samples

were collected from a depth of 5 feet below the surface of the water using a peristaltic pump and dedicated tubing. Duplicate water samples from each location were analyzed for asbestos content at 2 different laboratories using EPA Methods 100.1 (TEM, Incorporated) and 100.2 (EMS Laboratories). The analytical results showed that no asbestos structures were counted by either laboratory using the referenced analytical methods. Copies of the laboratory reports are included in Appendix C.

Based upon the above analysis, no additional sampling of the bottom sediments of the Borrow Pit is warranted.

Tables

TABLE 1
SEDIMENT SAMPLING DATA

Johns Manville, Waukegan, IL
June 18 and 19, 2002

Basin	Sample Name	Date	Time	Water Depth (ft.)	Sludge Thickness (ft.)
Pumping Lagoon	SS-PL-1A	6/18/02	1320	13.8	1.2
	SS-PL-1B	6/18/02	1330	13.8	4.2
	SS-PL-1C	6/18/02	1345	12.9	4.1
Industrial Canal	SS-IC-2A	6/18/02	1045	14.5	1.6
	SS-IC-2B	6/18/02	1050	15.8	3.2
	SS-IC-2C	6/18/02	1055	15.1	1.9
	SS-IC-3A	6/18/02	1040	15.1	0.5
	SS-IC-3B	6/18/02	1005	16.8	>3.3
	SS-IC-3C	6/18/02	1000	15.1	0.5
	SS-IC-4A	6/18/02	945	15.5	3.4
	SS-IC-4B	6/18/02	1010	17.5	2.9
	SS-IC-4C	6/18/02	1025	13.8	1.1
Collection Basin	SS-CB-5A	6/19/02	1035	4.5	NA
	SS-CB-5B	6/19/02	1025	5.1	NA
	SS-CB-5C	6/19/02	1015	4.7	NA
	SS-CB-6A	6/19/02	1050	4.5	NA
	Field Duplicate (CB-6A)	6/19/02	1050	4.5	NA
	SS-CB-6B	6/19/02	1045	5.0	NA
	SS-CB-6C	6/19/02	1040	5.0	NA
Borrow Pit	SS-BP-7A	6/18/02	1445	13.2	1.8
	SS-BP-7B	6/18/02	1435	12.1	3.9
	SS-BP-7C	6/18/02	1425	11.3	1.7

TABLE 2
SEDIMENT SAMPLE ANALYTICAL RESULTS

Asbestos Content

Johns Manville, Waukegan, Illinois

June 18 and 19, 2002

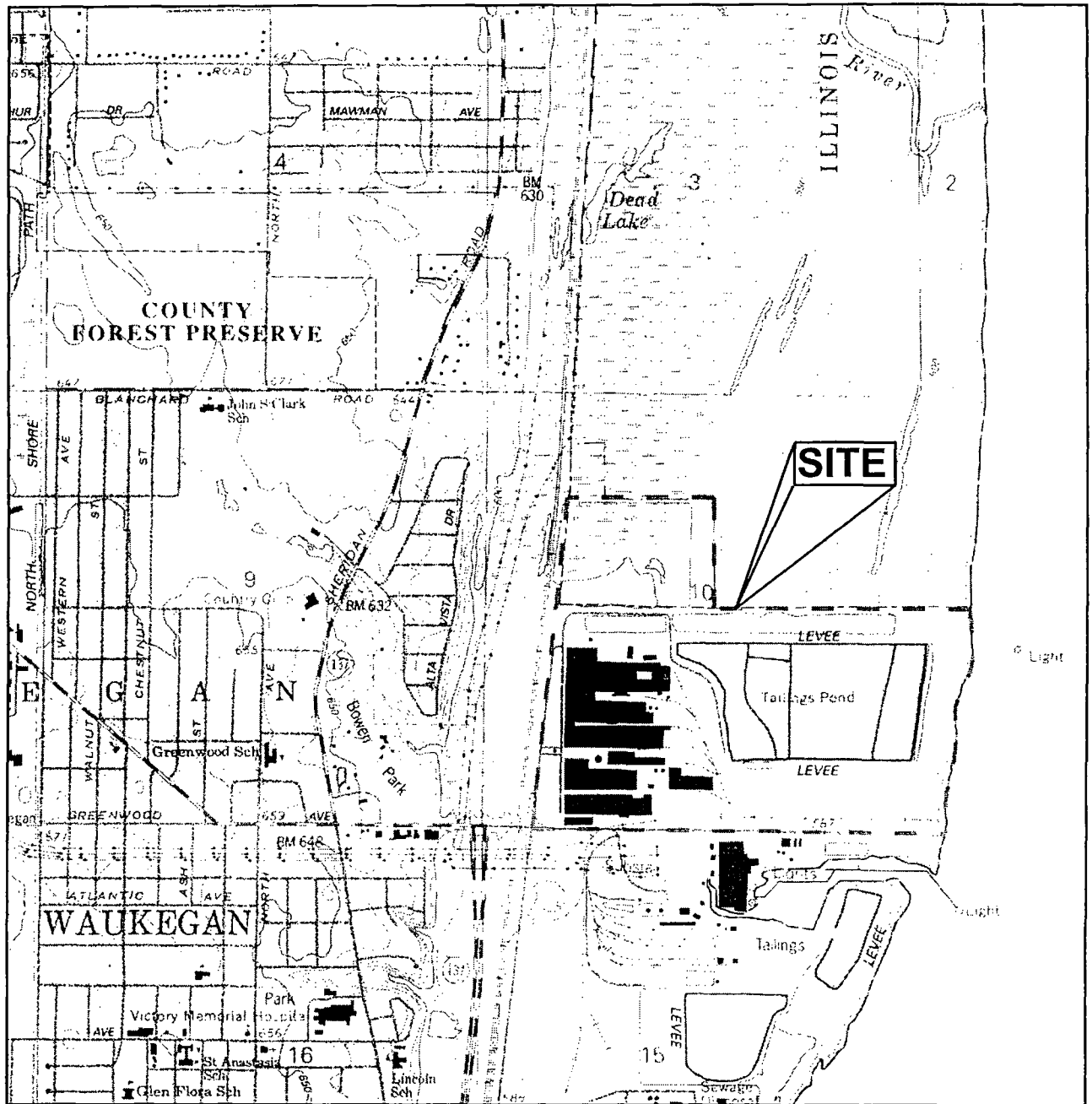
All concentrations are reported in % asbestos

Basin	Sample Name	% Chrysotile	% Amosite	% Crocidolite	% Total Asbestos
Pumping Lagoon	SS-PL-1A	4.4	2.4	ND	6.8
	SS-PL-1B	2.5	2.2	ND	4.7
	SS-PL-1C	<0.25	ND	ND	<0.25
Industrial Canal	SS-IC-2A	3.4	ND	ND	3.4
	SS-IC-2B	3.9	1.7	ND	5.6
	SS-IC-2C	4.0	1.7	ND	5.7
	SS-IC-3A	3.5	ND	ND	3.5
	SS-IC-3B	3.3	1.1	ND	4.4
	SS-IC-3C	1.0	ND	ND	1.0
	SS-IC-4A	3.2	ND	ND	3.2
	SS-IC-4B	1.0	ND	ND	1.0
	SS-IC-4C	1.9	ND	ND	1.9
Collection Basin	SS-CB-5A	1.9	ND	<0.25	1.9
	SS-CB-5B	2.0	1.0	ND	3.0
	SS-CB-5C	1.4	ND	<1.0	1.4
	SS-CB-6A	1.1	ND	ND	1.1
	SS-CB-6B	ND	ND	ND	ND
	SS-CB-6C	0.4	ND	ND	0.4
Borrow Pit	SS-BP-7A	<0.25	ND	ND	<0.25
	SS-BP-7B	<0.25	ND	ND	<0.25
	SS-BP-7C	ND	ND	ND	ND

"<" symbol indicates that asbestos was detected, but not quantifiable above the displayed detection limit.

ND: asbestos not detected in the sample

Figures



SITE LOCATION MAP

0 2000 FT.

Site Boundary

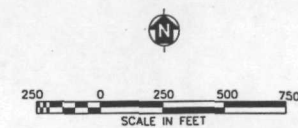
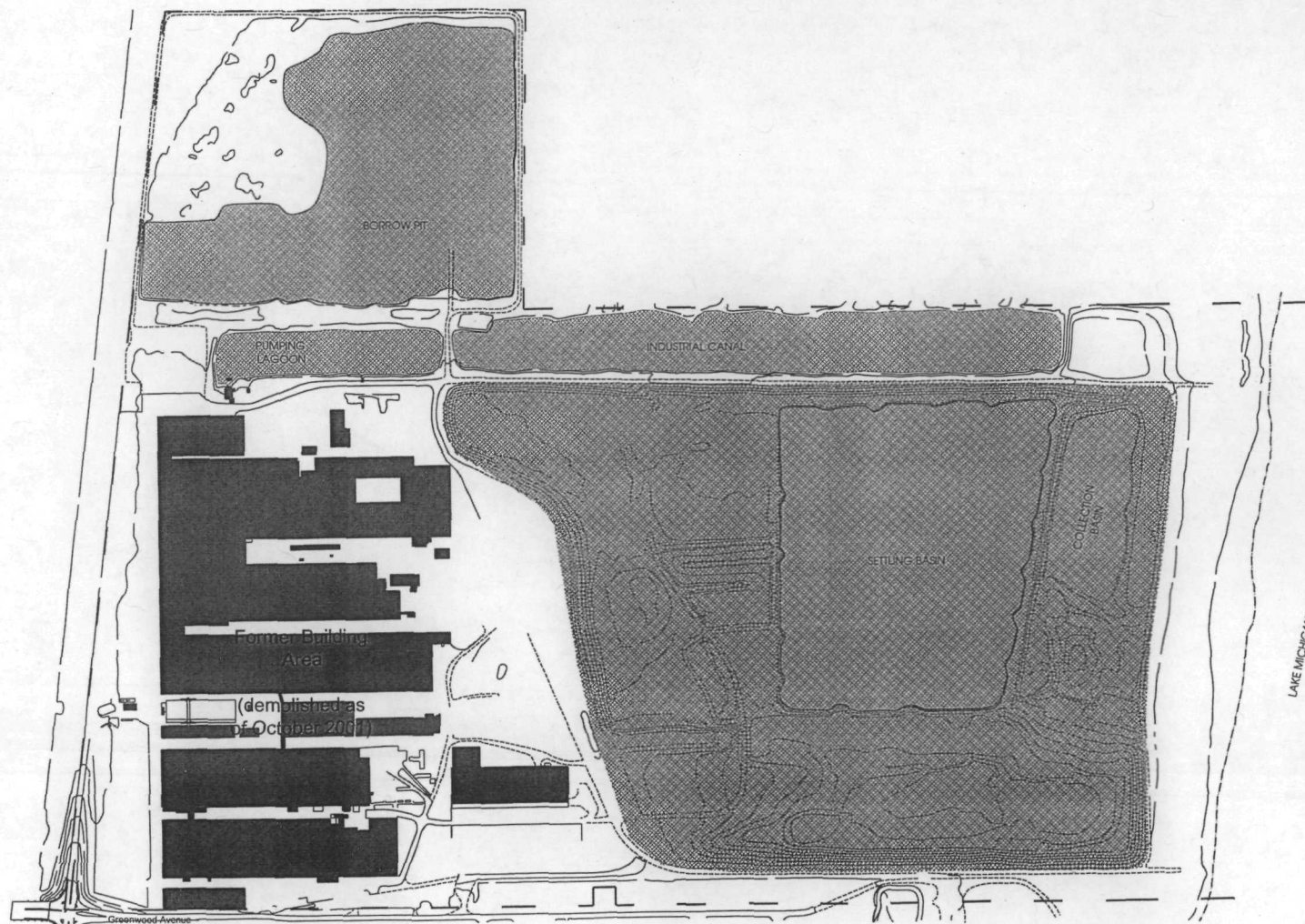
BASE MAP FROM 1993 USGS
7.5 MINUTE NE / 4 ZION 15' QUADRANGLE
ZION, ILLINOIS

Johns Manville

**1871 NORTH PERSHING ROAD
WAUKEGAN, ILLINOIS**

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Figure 1

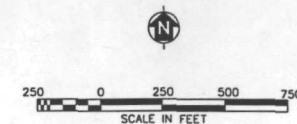
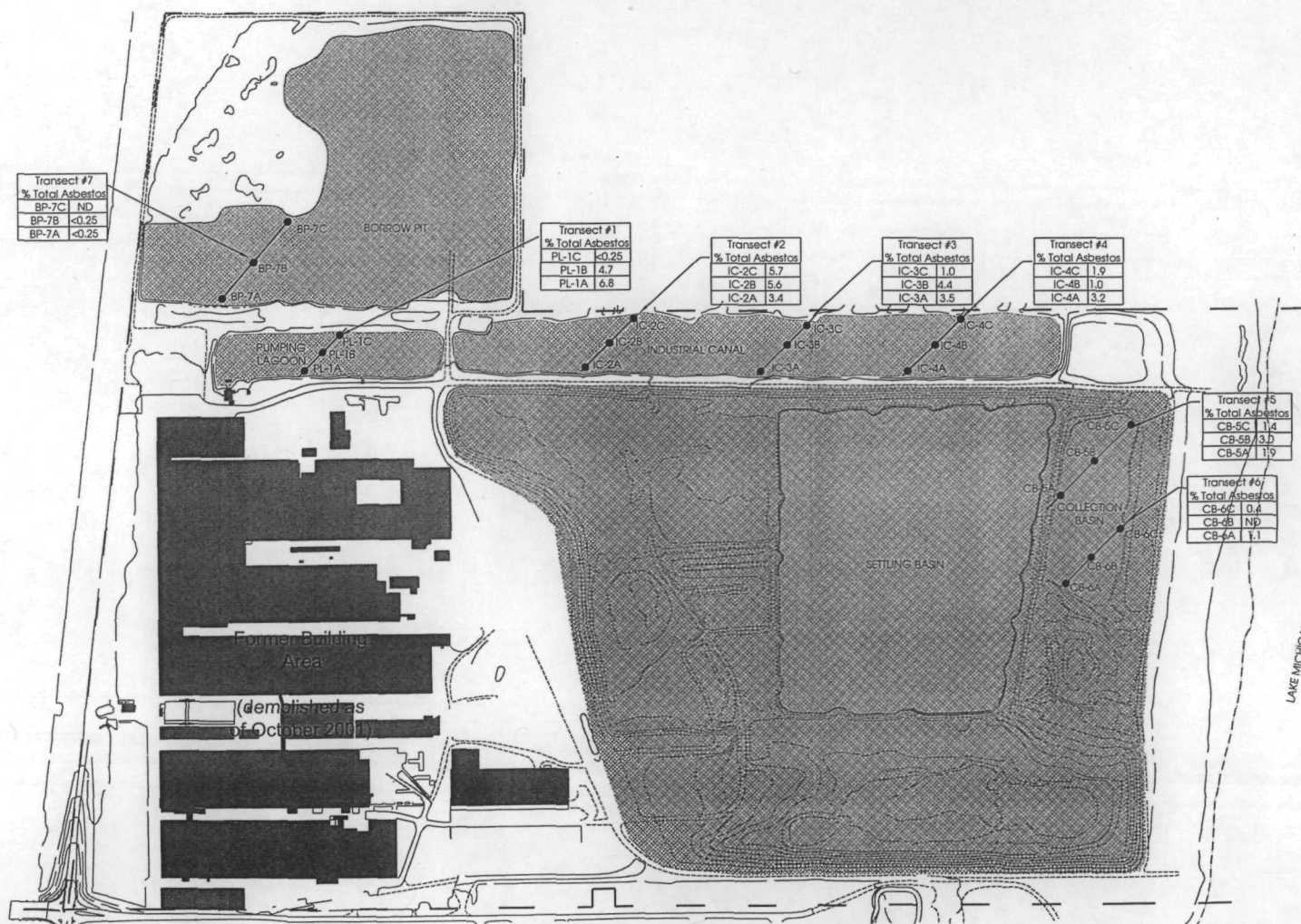


— — Property Line

JOHNS MANVILLE, WAUKEGAN, ILLINOIS
GENERAL PROPERTY MAP

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Figure 2

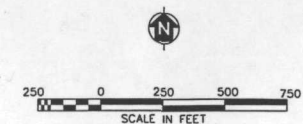
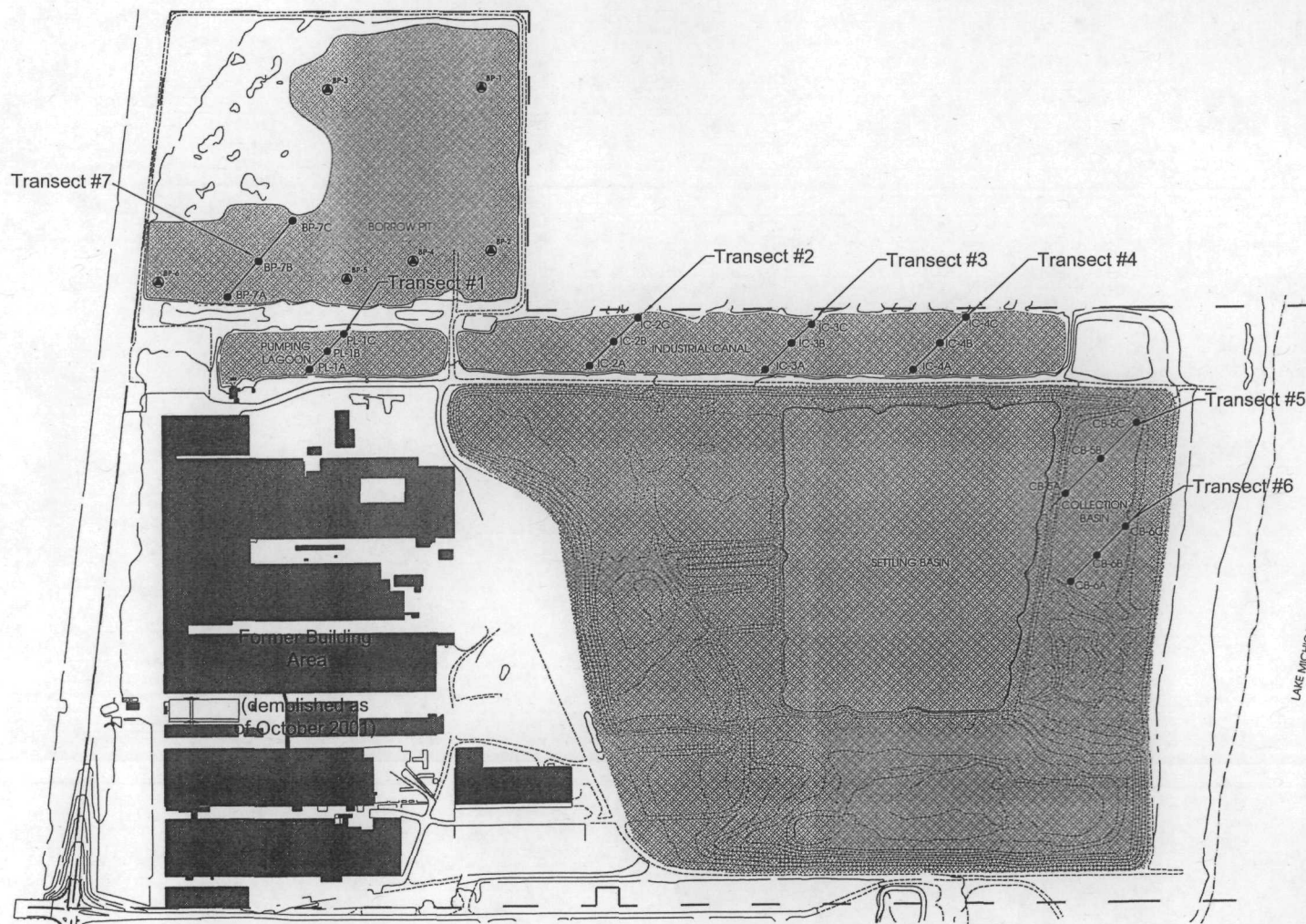


- Property Line
- Transect Line
- PL-1A Sediment Sample Location

JOHNS MANVILLE, WAUKEGAN, ILLINOIS
Sediment Sample Results

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Figure 4



- — Property Line
- — Transect Line
- PL-1A Sediment Sample Location
- BP-1 Borrow Pit Water Sample Location

JOHNS MANVILLE, WAUKEGAN, ILLINOIS
Borrow Pit Water Sample Locations

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Figure 5

APPENDICES

Appendix A

Sampling Plans and Amendments

June 11, 2002

FILE COPY
COPY

Mr. Brad Bradley
Remedial Project Manager
United States Environmental Protection Agency
Region 5
77 West Jackson Boulevard
Chicago, Illinois 60604-3590

Subject: Sediment Sampling; Johns Manville; 1871 North Pershing Road, Waukegan, Illinois 60087

Dear Mr. Bradley:

As we have discussed, the sediment sampling proposed for the Collection Basin, Industrial Canal, Pumping Lagoon, and Borrow Pit is scheduled to be completed on June 18 and 19, 2002. Sampling and analysis of the sediment will be completed in accordance with the following documents:

1. Sediment Sampling and Analysis Plan, letter from Johns Manville to United States Environmental Protection Agency (U.S. EPA), dated February 14, 2002.
2. Letter from U.S. EPA to Johns Manville, dated April 29, 2002.
3. Letter of Transmittal from LFR to U.S. EPA, U.S. EPA Region 1 Standard Operating Procedure for Screening Analysis of Soil and Sediment Samples for Asbestos Content, dated May 6, 2002.

Additionally, based upon verbal communication from U.S. EPA, a slight modification has been made to the location of the transect in the Borrow Pit. The attached transect location map reflects that change.

If you have any questions or concerns, please contact me at (847) 695-8855, extension 101.

Sincerely,



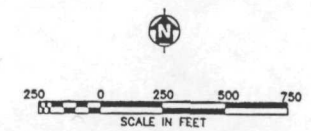
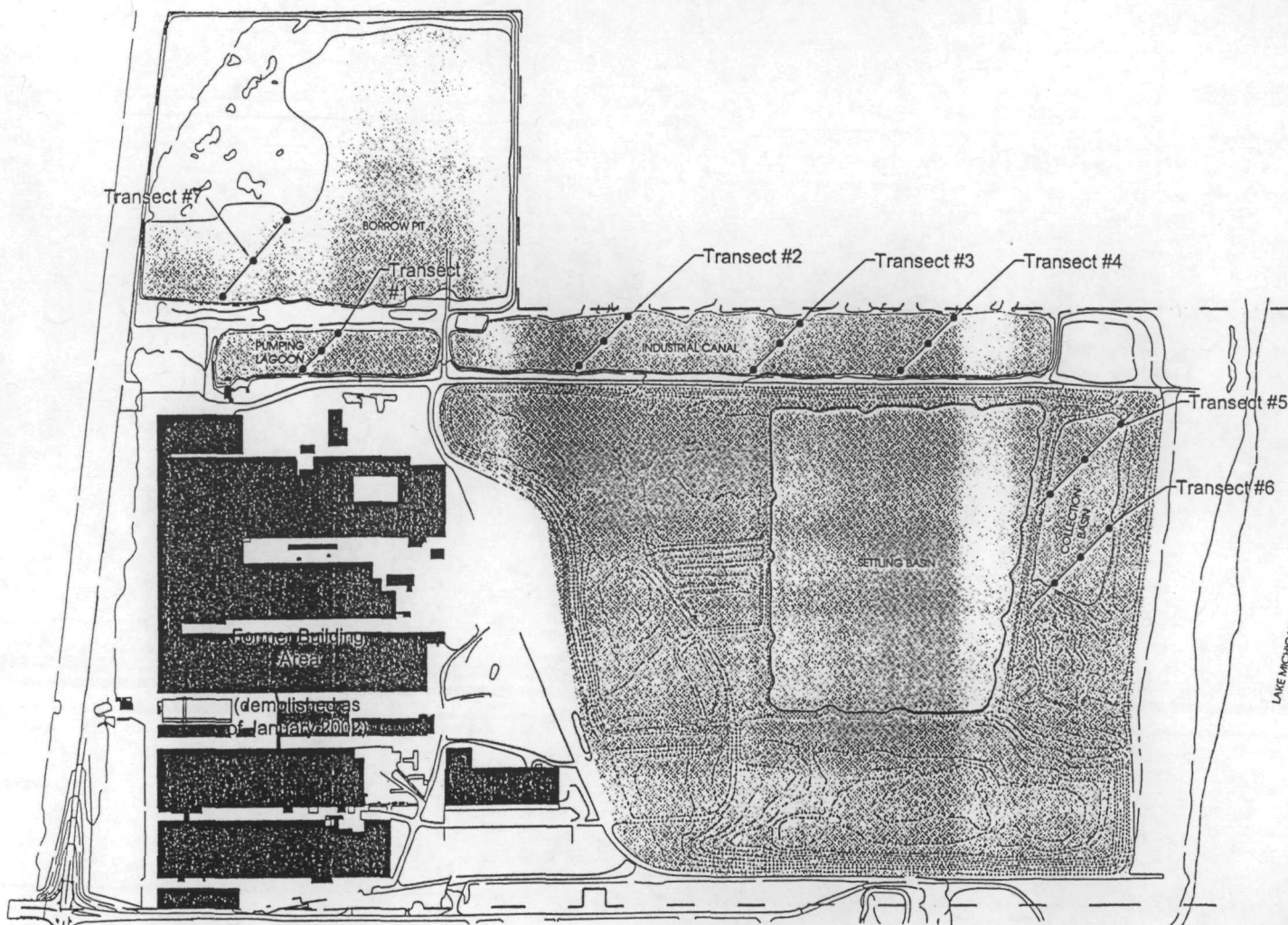
William A. Bow, C.P.G.
Principal, Operations Manager

Attachment

cc: Ann Coyle/USEPA
Sandra Bron/IEPA
Don Gimbel/IEPA
Elizabeth Wallace/Office of the Illinois Attorney General
W. Dennis Clinton/JM
Bruce D. Ray/JM
Edward P. Kenney/Sidley & Austin
David M. Peterson/David M. Peterson, PE, PC

Figure 1

**Revised Transect Lines and
Sediment Sample Location Map**



- — Property Line
- — Transect Line
- Proposed Sample Location

JOHNS MANVILLE, WAUKEGAN, ILLINOIS
 Proposed Transect Lines
 and Sediment Sample Locations
 Revised: June 11, 2002



Figure 1

May 6, 2002

FILE COPY

Mr. Brad Bradley
Project Manager
United States Environmental Protection Agency Region 5
77 West Jackson Boulevard
Chicago, IL 60604-3507

Subject: Letter of Transmittal; U.S. EPA Region 1 Standard Operating Procedure for Screening Analysis of Soil and Sediment Samples for Asbestos Content; Second Explanation of Significant Differences; Johns Manville, Waukegan, Illinois

Dear Mr. Bradley:

Enclosed is a copy of the U.S. EPA Region 1 Standard Operating Procedure for Screening Analysis of Soil and Sediment Samples for Asbestos Content. The Sediment Sampling and Analysis Plan prepared by Johns Manville (JM) pursuant to the Second Explanation of Significant Differences and dated February 14, 2002, proposed that various sediments located in the Industrial Canal, Pumping Lagoon, and Collection Basin be tested for the presence of asbestos. The attached protocols, as developed by U.S. EPA Region 1, represent to our knowledge, the best available technology for "visually estimating percentage levels of asbestos in a soil or sediment sample" (see Summary statement in protocol).

Please note that an addendum from EMSL Analytical, Inc. has also been attached. EMSL will perform the analysis in accordance with the method modifications in the addendum. The purpose of the EMSL modification to the Region 1 protocol is to capture and analyze (for asbestos) various fines that pass through the sieves used to sort the various size fractions in the original sample. The original Region 1 protocol called for discarding the finest size fraction resulting from washing the sample. This would result in discarding some unknown volume of the asbestos fibers potentially present in the sample. The EMSL modification calls for the "fine silt material" to be dried and analyzed for the presence of asbestos, thereby analyzing all size fractions of the original sample.

If you have any questions, please contact me at (847) 696-8855, extension 101.

Sincerely,



William A. Bow C.P.G.
Principal

Attachment

cc: Denny Clinton

SOP:EIA-INGASED2.SOP
Asbestos in Sediments/Soils
1/11/99
Page 1 of 8

EPA, Region I

Standard Operating Procedure for the
Screening Analysis of Soil and Sediment
Samples for Asbestos Content

Prepared for: Office of Environmental Measurement
And Evaluation
U.S. EPA, Region I

Revised by: Scott Clifford, Chemist, Investigations
And Analysis Unit, OEME

Approved by: Agnes VanLangenhove, Ph.D., QA Officer,
Investigations and Analysis Unit, OEME

Approved by: Robert E. Maxfield, Manager, Investigations
And Analysis Unit, OEME

SOP:EIA-INGASED2.SOP
Asbestos in Sediments/Soils
1/11/99
Page 2 of 8

1.0 Summary:

This SOP details the sample analysis protocol for determining the asbestos content of soil and sediment samples. Sample clean up with a sieve washing is followed by stereo microscope and polarized light microscope examination. This protocol is a semi-quantitative method, used only for visually estimating percentage levels of asbestos in a soil or sediment sample.

2.0 Purpose:

To ensure that the protocols for analysis of asbestos in sediment/soil samples are consistently applied by all analysts.

3.0 Scope and Application:

This protocol is not a "reference" method. It was developed out of necessity to facilitate finding asbestos fibers in a soil or mud (sediment) sample that does not contain any obvious asbestos fibers or asbestos-containing building or product materials when examined dry (or wet) using a stereo microscope at 10X or 20X magnification. It has been used for asbestos content in order to help delineate contaminated areas. It has proven to be an extremely sensitive method capable of finding very small amounts of asbestos fibers in a soil or sediment matrix.

Glassware or other materials, supplies, etc., mentioned below in the procedures are those being used in this laboratory. Other materials may be substituted as long as the primary purpose of the protocol is followed, namely: to find asbestos fibers in the sample. Identification of fibrous components is accomplished by the routine Polarized Light Microscopy (PLM) (with dispersion staining) method. (See EPA approved method references on

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Asbestos in Sediments/Soils

1/11/99

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page 6.)

4.0 Definitions:

PLM - Polarized Light Microscopy

5.0 Health and Safety Warnings:

Asbestos fibers can have serious effects on your health if inhaled. Sample containers should be initially opened in the HEPA filtered hood. Care must be taken when handling any unknown samples to prevent airborne asbestos.

ANALYTICAL PROCEDURES

1. A representative portion of the sample is removed from the sample container after thoroughly mixing for homogeneity. Because asbestos fibers usually cannot be seen because of the composition of the sample matrix such as the dirt, sand, mud, vegetation, water, etc., steps must be taken to clean up the sample to the point where the asbestos fibers, if any, may be seen using the stereomicroscope at 10X to 20 X magnification. A stereomicroscope is mandatory for this protocol.
2. To eliminate interfering particles, a 16mm ID by 150mm long, good quality PYREX or KIMAX test tube (not a fragile disposable tube) is used to remove portions of the well-mixed soil/sediment sample from several places in the sample container by pushing it into the sample to accumulate a sample depth of about 2.5 inches (65mm) in the test tube. A glass or plastic stirring rod is used to push the sample down into the tube and fiber-free (tap) water is added for shaking purposes. The soil and water mixture is shaken vigorously to loosen and separate the fines and other components of the sample and the contents of the test tube are then poured into a 3 inch ID, 60 mesh (250

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SOP:EIA-INGASED2.SOP

Asbestos in Sediments/Soils

1/11/99

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micrometers) sieve. This serves to eliminate, or greatly reduce, colloidal material, fine sand, silt and other non-fibrous particulates from the sample. More water is added to the tube, shaken and dumped into the sieve. Repeat this step until the tube is clean. The sample in the sieve is then rinsed until clean (clear water running through the sieve) with a fairly fine, pressurized stream of water from a plastic wash bottle.

All of the material remaining in the sieve is then washed from the sieve screen using a stream of water from the rinse bottle into a square plastic weighing dish of about 100ml liquid capacity. Use just enough water to completely cover the sample in the dish about 1/8th inch or so for examination with the stereo microscope.

After the cleaned sample is transferred to the weighing dish for examination, thoroughly rinse the sieve and test tube under running tap water (preferably aerated to minimize splashing) and carryover will not be a problem from sample to sample. It is a good idea to carry out all washing of the sample fines over a plastic dishpan or other container set into the sink basin in order to capture the fines and keep them from clogging the sink drain trap. After a settling period, the overlying water may be poured off and the fines/mud disposed of separately.

NOTE:

Since the purpose of the test is to find out if the soil or mud sample contains a significant amount of asbestos (>1%) that can be identified using the PLM technique, all portions of the sample are to be examined except those fines which pass through the sieve.

3. After examining them for asbestos fibers, floating pieces of organic material such as roots, sticks, leaves, etc., may be removed to get a better view of the rest of the sample in the dish. Frequently, root structures found in surface soils will trap asbestos fibers during the shaking process and are a

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good place to look for the fibers. The sample is then carefully and systematically examined under the stereo microscope at 10X-20X magnification for visible asbestos fibers and fiber bundles. A good, bright, focused light source such as a Nicholas transformer-base external illuminator is very helpful here. The fibers tend to stand out, shine, flash, etc., in the clean water matrix. Poking and stirring the sample with forceps and/or dissecting needles will help to locate the fibers. If no fibers are seen, gently shaking the weighing dish to redistribute particles will sometimes turn up previously hidden fibers when scanning the sample a second time. Suspect fibers are removed with sharp forceps and placed upon a clean microscope slide.

4. After picking as many suspect fibers or other material from the sample as necessary to determine its content, the slide preparation is allowed to dry and prepared for PLM analysis using an appropriate high-dispersion refractive index liquid and coverslip.

5. Next, the slide preparation is examined with a polarized light microscope (PLM) with dispersion staining to identify any fibers found. Standard, EPA approved PLM procedures are used to identify any asbestos fibers found as to specific type and form. The identification of asbestos using PLM is rapid and unequivocal due to the unique optical crystallographic properties of morphology, refractive indices, elongation, angle of extinction, dispersion and birefringence. Slide preps are examined for each sample with suspect fibers to confirm the presence of asbestos.

6. If asbestos fibers are identified, return to the sieved sample under the stereo microscope, observe the remaining asbestos fibers and bundles of fibers, and make a visual estimate of the percentage asbestos content in the whole sample including the material previously washed through the sieve. (This is all based on asbestos fibers seen using 10X to 20X magnification under the stereomicroscope.) Obviously, many of the finest fibers pass through the sieve and the finest ones remaining can't be seen at 20X magnification, but, this protocol is not meant to be used as a quantitative method. It is useful, however, to determine whether or not the soil or sediment is contaminated with significant amounts of asbestos. (> than 1 % by volume).

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7. As a rule, if asbestos fibers and/or fiber bundles can't be found relatively quickly and easily (one to two minutes) in the cleaned up sample under the stereo microscope, the percent asbestos content is most likely less than 0.1% and certainly less than 1.0% ! One should be able to find asbestos fibers in a sample containing more than 1.0% in a few seconds up to a one minute examination under the stereo microscope.
8. Usually, as much of the original sample as possible is returned to the original sample container. If saving the fines is required for further examination by other methods, use individual beakers to catch the sieve washings containing the sand and silt components.
9. The sample containers are then resealed before storage, disposal or return to the organization that requested the analyses.
10. If further information is required on this protocol or if anyone has found a better way to find and estimate asbestos fibers in muds or soils, please contact:

Scott Clifford or Dan Boudreau
USEPA, REGION 1
~~60 Westview St.~~ // Technology Drive
Lexington, MA 02421 Telephone: ~~(781) 860-4300~~
N. Chelmsford, MA 01863 (617) 918-8631

APPROVED EPA BULK ANALYSIS PROTOCOL:

40 CFR PART 763, SUBPART F, APPENDIX A

or, "ASBESTOS IDENTIFICATION"- Walter C. McCrone, 1987, McCrone Research Institute, Chicago, Ill.

See also, "Asbestos Content In Bulk Insulation Samples: Visual Estimates and Weight Composition"- US EPA, EPA-560/5-88-011, September, 1988.

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7.0 Analytical Procedure Addendum for More Accurate Quantitation:

. ADDENDUM TO "PROTOCOL FOR SCREENING SOIL AND SEDIMENT SAMPLES FOR ASBESTOS CONTENT USED BY THE U.S. ENVIRONMENTAL PROTECTION AGENCY, REGION I LABORATORY"

Addendum dated: August 1997

This addendum can be used to more accurately quantitate the volume of asbestos in soil and sediment samples. It is meant to give the analyst a good visual estimate of the fine materials volume which pass through the mesh sieve relative to the original sample volume analyzed. It must be used in conjunction with the above mentioned protocol.

Sample Preparation Analytical Procedure:

1. Transfer a well mixed portion of homogenized soil into the plastic weigh dish. Cover the bottom of the dish with a thin (1cm) layer. Quantitatively transfer the soil/sediment into the test tube using a wide mouth funnel. Fiber-free (tap) water can be used to help wash fines into the test tube. A glass or plastic stirring rod is used to push the sample down into the test tube end and to break-up any soil clumps. Wash the test tube sides down with a stream of water. Let the soil/water mix settle such that the volume of material in the test tube can be measured. Measure from the bottom of the test tube to the top of the settled soil with a ruler and record the value (i.e., 4.5cm).

After the soil volume measurement, continue the analytical procedure

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(i.e., shake vigorously to loosen and separate the fines, pour contents into sieve for clean-up, transfer sample component left in sieve to weigh dish for examination, etc.)

After complete examination and determination of asbestos content of the sample portion in the weigh dish which did not pass through the sieve (using stereo microscope and PLM), the sample in the weigh dish is quantitatively transferred back into the test tube and allowed to settle. After settling, the volume of material in the test tube is again measured and recorded (i.e., 2.0cm).

Determine the asbestos content of the sample as follows:

% asbestos in sample portion which did not pass through the sieve	X	Volume of sample which did not pass through <u>sieve</u> Initial sample volume
---	---	---

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**ANALYTICAL, INC.**

Asbestos – Lead – Environmental – Materials & Indoor Air Analysis



Corporate Office & Lab
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**EPA PROTOCOL FOR SCREENING SOIL AND SEDIMENT SAMPLES
FOR ASBESTOS CONTENT USED BY THE US ENVIRONMENTAL
PROTECTION AGENCY, REGION 1 LABORATORY
REVISED MAY 24,1994
EMSL MODIFIED (SEPT 1999) TO PROVIDE EITHER QUALITATIVE
OR QUANTITATIVE RESULTS.**

1. Transfer soil sample into a 500ml or larger jar with a screw cap cover and fill with deionized water up to $\frac{3}{4}$ capacity and shake well, loosening the soil sample.
2. Sieve the soil / water mixture through a 2mm sieve with deionized water. Sieve again through a 250 micron sieve. Collect the fine silt material in a basin.
3. Transfer the two sieved portions from the screens to drying dishes and dry in a convection oven at around 150°C.
4. Filter the fine silt material and oven dry.
5. Analyze with PLM (EPA 600 method) each of the three subsamples.
6. For a qualitative analysis report only as >1% or <1%.
7. For a quantitative analysis each of the three samples are weighed dry prior to PLM analysis and a quantitative result is generated through a weighted average calculation.

Peter Frasca Ph.D.
Director
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procedures are recommended to augment current federal requirements:

- Designate a separate area for asbestos waste disposal. Provide a record for future landowners that asbestos waste has been buried there and that it would be hazardous to attempt to excavate that area. (Future regulations may require property deeds to identify the location of any asbestos wastes and warn against excavation.)

- Prepare a separate trench to receive asbestos wastes. The size of the trench will depend upon the quantity and frequency of asbestos waste delivered to the disposal site. The trenching technique allows application of soil cover without disturbing the asbestos waste containers. The trench should be ramped to allow the transport vehicle to back into it, and the trench should be as narrow as possible to reduce the amount of cover required. If possible, the trench should be aligned perpendicular to prevailing winds.

- Place the asbestos waste containers into the trench carefully to avoid breaking them. Be particularly careful with plastic bags because when they break under pressure asbestos particles can be emitted.

- Completely cover the containerized waste within 24 hours with a minimum of 6 inches of nonasbestos material. Improperly containerized waste is a violation of the NESHAPs and EPA should be notified.

However, if improperly containerized waste is received at the disposal site, it should be covered immediately after unloading. Only after the wastes, including properly containerized wastes, are completely covered, can the wastes be compacted or other heavy equipment run over it. During compacting, avoid exposing wastes to the air or tracking asbestos material away from the trench.

- For final closure of an area containing asbestos waste, cover with at least an additional 30 inches of compacted nonasbestos material to provide a 36-inch final cover. To control erosion of the final cover, it should be properly graded and vegetated. In areas of the United States where excessive soil erosion may occur or the frost line exceeds 3 feet, additional final cover is recommended. In desert areas where vegetation would be difficult to maintain, 3–6 inches of well graded crushed rock is recommended for placement on top of the final cover.

Controlling public access. Under the current NESHAPs regulation, EPA does not require that a landfill used for asbestos disposal use warning signs or fencing if it meets the requirement to cover asbestos wastes. However, under RCRA, EPA requires that access be controlled to prevent exposure of the public to potential health and safety hazards at the disposal site. Therefore, for liability protection of operators of landfills that handle asbestos, fencing and warning signs are recommended to control public access when natural barriers do not exist. Access to a landfill should be limited to one or two entrances with gates that can be locked when left unattended. Fencing should be installed around the perimeter of the disposal site in a manner adequate to deter access by the general public. Chain-link fencing, 6-ft high and topped with a barbed wire guard, should be used. More specific fencing requirements may be specified by local regulations. Warning signs should be displayed at all entrances and at intervals of 330 feet or less along the property line of the landfill or perimeter of the sections where asbestos waste is deposited. The sign should read as follows:

ASBESTOS WASTE DISPOSAL SITE

BREATHING ASBESTOS DUST MAY CAUSE LUNG DISEASE AND CANCER

Recordkeeping. For protection from liability, and considering possible future requirements for notification on disposal site deeds, a landfill owner should maintain documentation of the specific location and quantity of the buried asbestos wastes. In addition, the estimated depth of the waste below the surface should be recorded whenever a landfill section is closed. As mentioned previously, such information should be recorded in the land deed or other record along with a notice warning against excavation of the area.

[52 FR 41897, Oct. 30, 1987, as amended at 62 FR 1834, Jan. 14, 1997]

Pt. 763, Subpt. E, App. E

APPENDIX E TO SUBPART E—INTERIM METHOD OF THE DETERMINATION OF ASBESTOS IN BULK INSULATION SAMPLES

SECTION 1, POLARIZED LIGHT MICROSCOPY

1.1 Principle and Applicability

Bulk samples of building materials taken for asbestos identification are first examined for homogeneity and preliminary fiber identification at low magnification. Positive identification of suspect fibers is made by analysis of subsamples with the polarized light microscope.

The principles of optical mineralogy are well established.^{1,2} A light microscope equipped with two polarizing filters is used to observe specific optical characteristics of a sample. The use of plane polarized light allows the determination of refractive indices along specific crystallographic axes. Morphology and color are also observed. A retardation plate is placed in the polarized light path for determination of the sign of elongation using orthoscopic illumination. Orientation of the two filters such that their vibration planes are perpendicular (crossed polars) allows observation of the birefringence and extinction characteristics of anisotropic particles.

Quantitative analysis involves the use of point counting. Point counting is a standard technique in petrography for determining the relative areas occupied by separate minerals in thin sections of rock. Background information on the use of point counting² and the interpretation of point count data³ is available.

This method is applicable to all bulk samples of friable insulation materials submitted for identification and quantitation of asbestos components.

1.2 Range

The point counting method may be used for analysis of samples containing from 0 to 100 percent asbestos. The upper detection limit is 100 percent. The lower detection limit is less than 1 percent.

1.3 Interferences

Fibrous organic and inorganic constituents of bulk samples may interfere with the identification and quantitation of the asbestos mineral content. Spray-on binder materials may coat fibers and affect color or obscure optical characteristics to the extent of masking fiber identity. Fine particles of other materials may also adhere to fibers to an

extent sufficient to cause confusion in identification. Procedures that may be used for the removal of interferences are presented in Section 1.7.2.2.

1.4 Precision and Accuracy

Adequate data for measuring the accuracy and precision of the method for samples with various matrices are not currently available. Data obtained for samples containing a single asbestos type in a simple matrix are available in the EPA report *Bulk Sample Analysis for Asbestos Content: Evaluation of the Tentative Method*.⁴

1.5 Apparatus

1.5.1 Sample Analysis

A low-power binocular microscope, preferably stereoscopic, is used to examine the bulk insulation sample as received.

- *Microscope*: binocular, 10–45X (approximate).
- *Light Source*: incandescent or fluorescent.
- *Forceps, Dissecting Needles, and Probes*
- *Glassine Paper or Clean Glass Plate*

Compound microscope requirements: A polarized light microscope complete with polarizer, analyzer, port for wave retardation plate, 360° graduated rotating stage, substage condenser, lamp, and lamp iris.

- *Polarized Light Microscope*: described above.
- *Objective Lenses*: 10X, 20X, and 40X or near equivalent.
- *Dispersion Staining Objective Lens* (optional)
- *Ocular Lens*: 10X minimum.
- *Eye-piece Reticle*: cross hair or 25 point Chalkley Point Array.
- *Compensator Plate*: 550 millimicron retardation.

1.5.2 Sample Preparation

Sample preparation apparatus requirements will depend upon the type of insulation sample under consideration. Various physical and/or chemical means may be employed for an adequate sample assessment.

- *Ventilated Hood* or negative pressure glove box.
- *Microscope Slides*
- *Coverslips*
- *Mortar and Pestle*: agate or porcelain. (optional)
- *Wylie Mill* (optional)
- *Beakers and Assorted Glassware* (optional)
- *Centrifuge* (optional)
- *Filtration apparatus* (optional)
- *Low temperature asher* (optional)

1.6 Reagents

1.6.1 Sample Preparation

- *Distilled Water* (optional)
- *Dilute CH₃COOH*: ACS reagent grade (optional)
- *Dilute HCl*: ACS reagent grade (optional)
- *Sodium metaphosphate (NaPO₃)₆* (optional)

1.6.2 Analytical Reagents

Refractive Index Liquids: 1.490–1.570, 1.590–1.720 in increments of 0.002 or 0.004.

- *Refractive Index Liquids for Dispersion Staining*: high-dispersion series, 1.550, 1.605, 1.630 (optional).
- *UICC Asbestos Reference Sample Set*: Available from: UICC MRC Pneumoconiosis Unit, Llandough Hospital,

Penarth, Glamorgan CF6 1XW, UK, and commercial distributors.

- *Tremolite-asbestos* (source to be determined)
- *Actinolite-asbestos* (source to be determined)

1.7 Procedures

NOTE: Exposure to airborne asbestos fibers is a health hazard. Bulk samples submitted for analysis are usually friable and may release fibers during handling or matrix reduction steps. All sample and slide preparations should be carried out in a ventilated hood or glove box with continuous airflow (negative pressure). Handling of samples without these precautions may result in exposure of the analyst and contamination of samples by airborne fibers.

1.7.1 Sampling

Samples for analysis of asbestos content shall be taken in the manner prescribed in Reference 5 and information on design of sampling and analysis programs may be found in Reference 6. If there are any questions about the representative nature of the sample, another sample should be requested before proceeding with the analysis.

1.7.2 Analysis

1.7.2.1 Gross Examination

Bulk samples of building materials taken for the identification and quantitation of asbestos are first examined for homogeneity at low magnification with the aid of a stereomicroscope. The core sample may be examined in its container or carefully removed from the container onto a glassine transfer paper or clean glass plate. If possible, note is made of the top and bottom orientation. When discrete strata are identified, each is treated as a separate material so that fibers are first identified and quantified in that layer only, and then the results for each layer are combined to yield an estimate of asbestos content for the whole sample.

1.7.2.2 Sample Preparation

Bulk materials submitted for asbestos analysis involve a wide variety of matrix materials. Representative subsamples may not be readily obtainable by simple means in heterogeneous materials, and various steps may be required to alleviate the difficulties encountered. In most cases, however, the best preparation is made by using forceps to sample at several places from the bulk material. Forcep samples are immersed in a refractive index liquid on a microscope slide, teased apart, covered with a cover glass, and observed with the polarized light microscope.

Alternatively, attempts may be made to homogenize the sample or eliminate interferences before further characterization. The selection of appropriate procedures is dependent upon the samples encountered and personal preference. The following are presented as possible sample preparation steps.

A mortar and pestle can sometimes be used in the size reduction of soft or loosely bound materials though this may cause matting of some samples. Such samples may be reduced in a Wylie mill. Apparatus should be clean and extreme care exercised to avoid cross-contamination of samples. Periodic checks of the particle sizes should be made during the grinding operation so as to preserve any fiber bundles present in an identifiable form. These procedures are not recommended for samples that contain amphibole minerals or vermiculite. Grinding of

amphiboles may result in the separation of fiber bundles or the production of cleavage fragments with aspect ratios greater than 3:1. Grinding of vermiculite may also produce fragments with aspect ratios greater than 3:1.

Acid treatment may occasionally be required to eliminate interferences. Calcium carbonate, gypsum, and bassanite (plaster) are frequently present in sprayed or trowelled insulations. These materials may be removed by treatment with warm dilute acetic acid. Warm dilute hydrochloric acid may also be used to remove the above materials. If acid treatment is required, wash the sample at least twice with distilled water, being careful not to lose the particulates during decanting steps. Centrifugation or filtration of the suspension will prevent significant fiber loss. The pore size of the filter should be 0.45 micron or less. Caution: prolonged acid contact with the sample may alter the optical characteristics of chrysotile fibers and should be avoided.

Coatings and binding materials adhering to fiber surfaces may also be removed by treatment with sodium metaphosphate.⁷ Add 10 mL of 10g/L sodium metaphosphate solution to a small (0.1 to 0.5 mL) sample of bulk material in a 15-mL glass centrifuge tube. For approximately 15 seconds each, stir the mixture on a vortex mixer, place in an ultrasonic bath and then shake by hand. Repeat the series. Collect the dispersed solids by centrifugation at 1000 rpm for 5 minutes. Wash the sample three times by suspending in 10 mL distilled water and recentrifuging. After washing, resuspend the pellet in 5 mL distilled water, place a drop of the suspension on a microscope slide, and dry the slide at 110° C.

In samples with a large portion of cellulosic or other organic fibers, it may be useful to ash part of the sample and view the residue. Ashing should be performed in a low temperature ashers. Ashing may also be performed in a muffle furnace at temperatures of 500° C or lower. Temperatures of 550° C or higher will cause

dehydroxylation of the asbestos minerals, resulting in changes of the refractive index and other key parameters. If a muffle furnace is to be used, the furnace thermostat should be checked and calibrated to ensure that samples will not be heated at temperatures greater than 550° C.

Ashing and acid treatment of samples should not be used as standard procedures. In order to monitor possible changes in fiber characteristics, the material should be viewed microscopically before and after any sample preparation procedure. Use of these procedures on samples to be used for quantitation requires a correction for percent weight loss.

1.7.2.3 Fiber Identification

Positive identification of asbestos requires the determination of the following optical properties.

- Morphology
- Color and pleochroism
- Refractive indices
- Birefringence
- Extinction characteristics
- Sign of elongation

Table 1-1 lists the above properties for commercial asbestos fibers. Figure 1-1 presents a flow diagram of the examination procedure. Natural variations in the conditions under which deposits of asbestiform minerals are formed will occasionally produce exceptions to the published values and differences from the UICC standards. The sign of elongation is determined by use of the compensator plate and crossed polars. Refractive indices may be determined by the Becke line test. Alternatively, dispersion staining may be used. Inexperienced operators may find that the dispersion staining technique is more easily learned, and should consult Reference 9 for guidance. Central stop dispersion staining colors are presented in Table 1-2. Available high-dispersion (HD) liquids should be used.

TABLE 1-1—OPTICAL PROPERTIES OF ASBESTOS FIBERS

Mineral	Morphology, color*	Refractive indices ^b		Birefringence	Extinction	Sign of elongation
		α	γ			
Chrysotile (asbestiform serpentine).	Wavy fibers. Fiber bundles have splayed ends and "kinks". Aspect ratio typically >10:1. Colorless ¹ , nonpleochroic.	1.493-1.560	1.517-1.562 ^c (normally 1.556).	.008	No fiber length.	+ (length slow)
Amosite (asbestiform grunerite).	Straight, rigid fibers. Aspect ratio typically >10:1. Colorless to brown, nonpleochroic or weakly so. Opaque inclusions may be present.	1.635-1.696	1.655-1.729 ^c (normally 1.696-1.710).	.020-.033	No fiber length.	+ (length slow)
Crocidolite (asbestiform riebeckite).	Straight, rigid fibers. Thick fibers and bundles common, blue to purple-blue in color. Pleochroic. Birefringence is generally masked by blue color.	1.654-1.701	1.668-1.717 ^c (normally close to 1.700).	.014-.016	No fiber length.	- (length fast)
Anthophyllite-asbestos.	Straight fibers and acicular cleavage fragments. ^d Some composite fibers. Aspect ratio <10:1. Colorless to light brown.	1.596-1.652	1.615-1.676 ^c	.019-.024	No fiber length.	+ (length slow)

TABLE 1-1—OPTICAL PROPERTIES OF ASBESTOC FIBERS—Continued

Mineral	Morphology, color ^a	Refractive indices ^b		Birefringence	Extinction	Sign of elongation
		α	γ			
Tremolite-actinolite-asbestos.	Normally present as acicular or prismatic cleavage fragments. ^c Single crystals predominate, aspect ratio <10:1. Colorless to pale green.	1.599–1.668	1.622–1.688 ^f	.023–.020	Oblique extinction, 10–20° for fragments. Composite fibers show extinction.	+ (length slow)

^aFrom reference 5; colors cited are seen by observation with plane polarized light.

^bFrom references 5 and 8.

^cFibers subjected to heating may be brownish.

^dFibers defined as having aspect ratio >3:1.

^e1 to fiber length.

^f1 to fiber length.

EC01AP92.017

TABLE 1-2—CENTRAL STOP DISPERSION STAINING COLORS_a

Mineral	RI Liquid	η_{\perp}	η_{\parallel}
Chrysotile	1.550 ¹⁰⁰	Blue	Blue-magenta
Amosite	1.680	Blue-magenta to pale blue.	Golden-yellow
	1.550 ¹⁰⁰	Yellow to white.	Yellow to white
Crocidolite ^b	1.700	Red magenta	Blue-magenta
	1.550 ¹⁰⁰	Yellow to white.	Yellow to white
Anthophyllite	1.605 ¹⁰⁰	Blue	Gold to gold-magenta
Tremolite	1.605 ^{100c}	Pale blue	Gold
Actinolite	1.605 ¹⁰⁰	Gold-magenta to blue.	Gold
	1.630 ^{100c}	Magenta	Golden-yellow

^aFrom reference 9.

^bBlue absorption color.

^cOblique extinction view.

1.7.2.4 Quantitation of Asbestos Content

Asbestos quantitation is performed by a point-counting procedure or an equivalent estimation method. An ocular reticle (cross-hair or point array) is used to visually superimpose a point or points on the microscope field of view. Record the number of points positioned directly above each kind of particle or fiber of interest. Score only points directly over asbestos fibers or nonasbestos matrix material. Do not score empty points for the closest particle. If an asbestos fiber and a matrix particle overlap so that a point is superimposed on their visual intersection, a point is scored for both categories. Point counting provides a determination of the area percent asbestos. Reliable conversion of area percent to percent of dry weight is not currently feasible unless the specific gravities and relative volumes of the materials are known.

For the purpose of this method, "asbestos fibers" are defined as having an aspect ratio greater than 3:1 and being positively identified as one of the minerals in Table 1-1.

A total of 400 points superimposed on either asbestos fibers or nonasbestos matrix material must be counted over at least eight different preparations of representative subsamples. Take eight forcep samples and mount each separately with the appropriate refractive index liquid. The preparation should not be heavily loaded. The sample should be uniformly dispersed to avoid overlapping particles and allow 25–50 percent empty area within the fields of view. Count 50 nonempty points on each preparation, using either

- A cross-hair reticle and mechanical stage; or
- A reticle with 25 points (Chalkley Point Array) and counting at least 2 randomly selected fields.

For samples with mixtures of isotropic and anisotropic materials present, viewing the sample with slightly uncrossed polars or the addition of the compensator plate to the polarized light path will allow simultaneous discrimination of both particle types. Quantitation should be performed at 100X or at the lowest magnification of the polarized light microscope that can effectively distinguish the sample components. Confirmation of the quantitation result by a second analyst on some percentage of analyzed samples should be used as standard quality control procedure.

The percent asbestos is calculated as follows:

$$\% \text{ asbestos} = (a/n) 100\%$$

where

a=number of asbestos counts,

n=number of nonempty points counted (400).

If a=0, report "No asbestos detected." If 0<a≤3, report "<1% asbestos".

The value reported should be rounded to the nearest percent.

1.8 References

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SECTION 2, X-RAY POWDER DIFFRACTION

2.1 Principle and Applicability

The principle of X-ray powder diffraction (XRD) analysis is well established.^{1,2} Any solid, crystalline material will diffract an impinging beam of parallel, monochromatic X-rays whenever Bragg's Law,

$$\lambda = 2d \sin \theta,$$

is satisfied for a particular set of planes in the crystal lattice, where

λ = the X-ray wavelength, Å;

d = the interplanar spacing of the set of reflecting lattice planes, Å; and

θ = the angle of incidence between the X-ray beam and the reflecting lattice planes.

By appropriate orientation of a sample relative to the incident X-ray beam, a diffraction pattern can be generated that, in most cases, will be uniquely characteristic of both the chemical composition and structure of the crystalline phases present.

Unlike optical methods of analysis, however, XRD cannot determine crystal morphology. Therefore, in asbestos analysis, XRD does not distinguish between fibrous and nonfibrous forms of the serpentine and amphibole minerals (Table 2-1). However, when used in conjunction with optical methods such as polarized light microscopy (PLM), XRD techniques can provide a reliable analytical method for the identification and characterization of asbestiform minerals in bulk materials.

For qualitative analysis by XRD methods, samples are initially scanned over limited diagnostic peak regions for the serpentine (~7.4 Å) and amphibole (8.2-8.5 Å) minerals (Table 2-2). Standard slow-scanning methods for bulk sample analysis may be used for materials shown by PLM to contain significant amounts of asbestos (>5-10 percent). Detection of minor or trace amounts of asbestos may require special sample preparation and step-scanning analysis. All samples that exhibit diffraction peaks in the diagnostic regions for asbestiform minerals are submitted to a full (5°-60° 2 θ ; 1° 2 θ /min) qualitative XRD scan, and their diffraction patterns are compared with standard reference powder diffraction patterns³ to verify initial peak assignments and to identify possible matrix interferences when subsequent quantitative analysis will be performed.

TABLE 2-1—THE ASBESTOS MINERALS AND THEIR NONASBESTIFORM ANALOGS

Asbestiform	Nonasbestiform
SERPENTINE	
Chrysotile	Antigorite, lizardite
AMPHIBOLE	
Anthophyllite asbestos	Anthophyllite
Cummingtonite-grunerite asbestos ("Amosite")	Cummingtonite-grunerite
Crocidolite	Riebeckite
Tremolite asbestos	Tremolite
Actinolite asbestos	Actinolite

TABLE 2-2—PRINCIPAL LATTICE SPACINGS OF ASBESTIFORM MINERALS *

Minerals	Principal d-spacings (Å) and relative intensities			JCPDS Powder diffraction file ³ number
Chrysotile	7.37 ₁₀₀	3.65 ₇₀	4.57 ₅₀	21-543 ^b
	7.36 ₁₀₀	3.66 ₈₀	2.45 ₅₅	25-645
	7.10 ₁₀₀	2.33 ₈₀	3.55 ₇₀	22-1162 (theoretical)
"Amosite"	8.33 ₁₀₀	3.06 ₇₀	2.756 ₇₀	17-745 (nonfibrous)
	8.22 ₁₀₀	3.060 ₈₅	3.25 ₇₀	27-1170 (UICC)
Anthophyllite	3.05 ₁₀₀	3.24 ₆₀	8.26 ₅₅	9-455
	3.06 ₁₀₀	8.33 ₇₀	3.23 ₅₀	16-401 (synthetic)
Anthophyllite	2.72 ₁₀₀	2.54 ₁₀₀	3.480 ₈₀	25-157
Crocidolite	8.35 ₁₀₀	3.10 ₅₅	2.720 ₅₅	27-1415 (UICC)
Tremolite	8.38 ₁₀₀	3.12 ₁₀₀	2.705 ₉₀	13-437 ^b
	2.706 ₁₀₀	3.14 ₉₅	8.43 ₄₀	20-1310 ^b (synthetic)
	3.13 ₁₀₀	2.706 ₆₀	8.44 ₄₀	23-666 (synthetic mixture with richterite)

*This information is intended as a guide, only. Complete powder diffraction data, including mineral type and source, should be referred to, to ensure comparability of sample and reference materials where possible. Additional precision XRD data on amosite, crocidolite, tremolite, and chrysotile are available from the U.S. Bureau of Mines.⁴

*Fibrosity questionable.

Accurate quantitative analysis of asbestos in bulk samples by XRD is critically dependent on particle size distribution, crystallite size, preferred orientation and matrix absorption effects, and comparability of standard reference and sample materials. The most intense diffraction peak that has been shown to be free from interference by prior qualitative XRD analysis is selected for quantitation of each asbestiform mineral. A "thin-layer" method of analysis^{5,6} is recommended in which, subsequent to comminution of the bulk material to $\sim 10 \mu\text{m}$ by suitable cryogenic milling techniques, an accurately known amount of the sample is deposited on a silver membrane filter. The mass of asbestiform material is determined by measuring the integrated area of the selected diffraction peak using a step-scanning mode, correcting for matrix absorption effects, and comparing with suitable calibration standards. Alternative "thick-layer" or bulk methods,^{7,8} may be used for semiquantitative analysis.

This XRD method is applicable as a confirmatory method for identification and quantitation of asbestos in bulk material samples that have undergone prior analysis by PLM or other optical methods.

2.2 Range and Sensitivity

The range of the method has not been determined.

The sensitivity of the method has not been determined. It will be variable and dependent upon many factors, including matrix effects (absorption and interferences), diagnostic reflections selected, and their relative intensities.

2.3 Limitations

2.3.1 Interferences

Since the fibrous and nonfibrous forms of the serpentine and amphibole minerals (Table 2-1) are indistinguishable by XRD techniques unless special sample preparation techniques and instrumentation are used,⁹ the presence of nonasbestiform serpentines and amphiboles in a sample will pose severe interference problems in the identification and quantitative analysis of their asbestiform analogs.

The use of XRD for identification and quantitation of asbestiform minerals in bulk samples may also be limited by the presence of other interfering materials in the sample. For naturally occurring materials the commonly associated asbestos-related mineral interferences can usually be anticipated. However, for fabricated materials the nature of the interferences may vary greatly (Table 2-3) and present more serious problems in identification and quantitation.¹⁰ Potential interferences are summarized in Table 2-4 and include the following:

- *Chlorite* has major peaks at 7.19 Å and 3.58 Å that interfere with both the primary (7.36 Å) and secondary (3.66 Å) peaks for chrysotile. Resolution of the primary peak to give good quantitative results may be possible when a step-scanning mode of operation is employed.
- *Halloysite* has a peak at 3.63 Å that interferes with the secondary (3.66 Å) peak for chrysotile.
- *Kaolinite* has a major peak at 7.15 Å that may interfere with the primary peak of chrysotile at 7.36 Å when present at concentrations of >10 percent. However, the

secondary chrysotile peak at 3.66 Å may be used for quantitation.

- *Gypsum* has a major peak at 7.5 Å that overlaps the 7.36 Å peak of chrysotile when present as a major sample constituent. This may be removed by careful washing with distilled water, or by heating to 300° C to convert gypsum to plaster of paris.
- *Cellulose* has a broad peak that partially overlaps the secondary (3.66 Å) chrysotile peak.⁸
- Overlap of major diagnostic peaks of the amphibole asbestos minerals, amosite, anthophyllite, crocidolite, and tremolite, at approximately 8.3 Å and 3.1 Å causes mutual interference when these minerals occur in the presence of one another. In some instances, adequate resolution may be attained by using step-scanning methods and/or by decreasing the collimator slit width at the X-ray port.

TABLE 2-3—COMMON CONSTITUENTS IN INSULATION AND WALL MATERIALS

A. Insulation materials

Chrysotile
 "Amosite"
 Crocidolite
 *Rock wool
 *Slag wool
 *Fiber glass
 Gypsum ($\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$)
 Vermiculite (micas)
 *Perlite
 Clays (kaolin)
 *Wood pulp
 *Paper fibers (talc, clay, carbonate fillers)
 Calcium silicates (synthetic)
 Opaques (chromite, magnetite inclusions in serpentine)
 Hematite (inclusions in "amosite")
 Magnesite
 *Diatomaceous earth

B. Spray finishes or paints

Bassanite
 Carbonate minerals (calcite, dolomite, vaterite)
 Talc
 Tremolite
 Anthophyllite
 Serpentine (including chrysotile)
 Amosite
 Crocidolite
 *Mineral wool
 *Rock wool
 *Slag wool
 *Fiber glass
 Clays (kaolin)
 Micas
 Chlorite
 Gypsum ($\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$)
 Quartz
 *Organic binders and thickeners
 Hydromagnesite
 Wollastonite
 Opaques (chromite, magnetite inclusions in serpentine)
 Hematite (inclusions in "amosite")
 *Amorphous materials—contribute only to overall scattered radiation and increased background radiation.

TABLE 2-4—INTERFERENCES IN XRD ANALYSIS
ASBESTIFORM MINERALS

Asbestiform mineral	Primary diagnostic peaks (approximate d-spacings, in Å)	Interference
Serpentine Chrysotile	7.4	Nonasbestiform serpentines (antigorite, lizardite)
	3.7	Chlorite Kaolinite Gypsum Chlorite Halloysite Cellulose
Amphibole "Amosite" Anthophyllite Crocidolite Tremolite	3.1	Nonasbestiform amphiboles (cummingtonite-grunerite, anthophyllite, riebeckite, tremolite)
	8.3	Mutual interferences Carbonates Talc Mutual interferences

• Carbonates may also interfere with quantitative analysis of the amphibole asbestos minerals, amosite, anthophyllite, crocidolite, and tremolite. Calcium carbonate (CaCO_3) has a peak at 3.035 Å that overlaps major amphibole peaks at approximately 3.1 Å when present in concentrations of >5 percent. Removal of carbonates with a dilute acid wash is possible; however, if present, chrysotile may be partially dissolved by this treatment.¹¹

• A major talc peak at 3.12 Å interferes with the primary tremolite peak at this same position and with secondary peaks of crocidolite (3.10 Å), amosite (3.06 Å), and anthophyllite (3.05 Å). In the presence of talc, the major diagnostic peak at approximately 8.3 Å should be used for quantitation of these asbestiform minerals.

The problem of intraspecies and matrix interferences is further aggravated by the variability of the silicate mineral powder diffraction patterns themselves, which often makes definitive identification of the asbestos minerals by comparison with standard reference diffraction patterns difficult. This variability results from alterations in the crystal lattice associated with differences in isomorphous substitution and degree of crystallinity. This is especially true for the amphiboles. These minerals exhibit a wide variety of very similar chemical compositions, with the result being that their diffraction patterns are characterized by having major (110) reflections of the monoclinic amphiboles and (210) reflections of the orthorhombic anthophyllite separated by less than 0.2 Å.¹²

2.3.2 Matrix Effects

If a copper X-ray source is used, the presence of iron at high concentrations in a sample will result in significant X-ray fluorescence, leading to loss of peak intensity along with increased background intensity and an overall decrease in sensitivity. This situation may be corrected by choosing an X-ray source other than copper; however, this is often accompanied both by loss of intensity and by decreased resolution of closely spaced reflections. Alternatively,

use of a diffracted beam monochromator will reduce background fluorescent radiation, enabling weaker diffraction peaks to be detected.

X-ray absorption by the sample matrix will result in overall attenuation of the diffracted beam and may seriously interfere with quantitative analysis. Absorption effects may be minimized by using sufficiently "thin" samples for analysis.^{5,13,14} However, unless absorption effects are known to be the same for both samples and standards, appropriate corrections should be made by referencing diagnostic peak areas to an internal standard^{7,8} or filter substrate (Ag) peak.^{5,6}

2.3.3 Particle Size Dependence

Because the intensity of diffracted X-radiation is particle-size dependent, it is essential for accurate quantitative analysis that both sample and standard reference materials have similar particle size distributions. The optimum particle size range for quantitative analysis of asbestos by XRD has been reported to be 1 to 10 μm .¹⁵ Comparability of sample and standard reference material particle size distributions should be verified by optical microscopy (or another suitable method) prior to analysis.

2.3.4 Preferred Orientation Effects

Preferred orientation of asbestiform minerals during sample preparation often poses a serious problem in quantitative analysis by XRD. A number of techniques have been developed for reducing preferred orientation effects in "thick layer" samples.^{7,8,15} However, for "thin" samples on membrane filters, the preferred orientation effects seem to be both reproducible and favorable to enhancement of the principal diagnostic reflections of asbestos minerals, actually increasing the overall sensitivity of the method.^{12,14} (Further investigation into preferred orientation effects in both thin layer and bulk samples is required.)

2.3.5 Lack of Suitably Characterized Standard Materials

The problem of obtaining and characterizing suitable reference materials for asbestos analysis is clearly recognized. NIOSH has recently directed a major research effort toward the preparation and characterization of analytical reference materials, including asbestos standards;^{16,17} however, these are not available in large quantities for routine analysis.

In addition, the problem of ensuring the comparability of standard reference and sample materials, particularly regarding crystallite size, particle size distribution, and degree of crystallinity, has yet to be adequately addressed. For example, Langer et al.¹⁸ have observed that in insulating matrices, chrysotile tends to break open into bundles more frequently than amphiboles. This results in a line-broadening effect with a resultant decrease in sensitivity. Unless this effect is the same for both standard and sample materials, the amount of chrysotile in the sample will be underestimated by XRD analysis. To minimize this problem, it is recommended that standardized matrix reduction procedures be used for both sample and standard materials.

2.4 Precision and Accuracy

Precision of the method has not been determined.
Accuracy of the method has not been determined.

2.5 Apparatus

2.5.1 Sample Preparation

Sample preparation apparatus requirements will depend upon the sample type under consideration and the kind of XRD analysis to be performed.

- *Mortar and Pestle*: Agate or porcelain.
- *Razor Blades*
- *Sample Mill*: SPEX, Inc., freezer mill or equivalent.
- *Bulk Sample Holders*
- *Silver Membrane Filters*: 25-mm diameter, 0.45- μ m pore size. Selas Corp. of America, Flotronics Div., 1957 Pioneer Road, Huntington Valley, PA 19006.
- *Microscope Slides*
- *Vacuum Filtration Apparatus*: Gelman No. 1107 or equivalent, and side-arm vacuum flask.
- *Microbalance*
- *Ultrasonic Bath or Probe*: Model W140, Ultrasonics, Inc., operated at a power density of approximately 0.1 W/mL, or equivalent.
- *Volumetric Flasks*: 1-L volume.
- *Assorted Pipettes*
- *Pipette Bulb*
- *Nonserrated Forceps*
- *Polyethylene Wash Bottle*
- *Pyrex Beakers*: 50-mL volume.
- *Desiccator*
- *Filter Storage Cassettes*
- *Magnetic Stirring Plate and Bars*
- *Porcelain Crucibles*
- *Muffle Furnace or Low Temperature Asher*

2.5.2 Sample Analysis

Sample analysis requirements include an X-ray diffraction unit, equipped with:

- *Constant Potential Generator; Voltage and mA Stabilizers*
- *Automated Diffractometer with Step-Scanning Mode*
- *Copper Target X-Ray Tube*: High intensity, fine focus, preferably.
- *X-Ray Pulse Height Selector*
- *X-Ray Detector* (with high voltage power supply): Scintillation or proportional counter.
- *Focusing Graphite Crystal Monochromator*; or *Nickel Filter* (if copper source is used, and iron fluorescence is not a serious problem).
- *Data Output Accessories*:
 - *Strip Chart Recorder*
 - *Decade Scaler/Timer*
 - *Digital Printer*
- *Sample Spinner* (optional).
- *Instrument Calibration Reference Specimen*: α -quartz reference crystal (Arkansas quartz standard, #180-147-00, Philips Electronics Instruments, Inc., 85 McKee Drive, Mahwah, NJ 07430) or equivalent.

2.6 Reagents

2.6.1 Standard Reference Materials

The reference materials listed below are intended to serve as a guide. Every attempt should be made to acquire pure reference materials that are comparable to sample materials being analyzed.

- *Chrysotile*: UICC Canadian, or NIEHS Plastibest. (UICC reference materials available from: UICC, MRC

Pneumoconiosis Unit, Llandough Hospital, Penarth, Glamorgan, CF61XW, UK).

- *Crocidolite*: UICC
- *Amosite*: UICC
- *Anthophyllite*: UICC
- *Tremolite Asbestos*: Wards Natural Science Establishment, Rochester, N.Y.; Cyprus Research Standard, Cyprus Research, 2435 Military Ave., Los Angeles, CA 90064 (washed with dilute HCl to remove small amount of calcite impurity); India tremolite, Rajasthan State, India.
- *Actinolite Asbestos*

2.6.2 Adhesive

Tape, petroleum jelly, etc. (for attaching silver membrane filters to sample holders).

2.6.3 Surfactant

1 percent aerosol OT aqueous solution or equivalent.

2.6.4 Isopropanol

ACS Reagent Grade.

2.7 Procedure

2.7.1 Sampling

Samples for analysis of asbestos content shall be collected as specified in EPA Guidance Document #C0090, *Asbestos-Containing Materials in School Buildings*.¹⁰

2.7.2 Analysis

All samples must be analyzed initially for asbestos content by PLM. XRD should be used as an auxiliary method when a second, independent analysis is requested.

NOTE: Asbestos is a toxic substance. All handling of dry materials should be performed in an operating fume hood.

2.7.2.1 Sample Preparation

The method of sample preparation required for XRD analysis will depend on: (1) The condition of the sample received (sample size, homogeneity, particle size distribution, and overall composition as determined by PLM); and (2) the type of XRD analysis to be performed (qualitative, quantitative, thin layer or bulk).

Bulk materials are usually received as inhomogeneous mixtures of complex composition with very wide particle size distributions. Preparation of a homogeneous, representative sample from asbestos-containing materials is particularly difficult because the fibrous nature of the asbestos minerals inhibits mechanical mixing and stirring, and because milling procedures may cause adverse lattice alterations.

A discussion of specific matrix reduction procedures is given below. Complete methods of sample preparation are detailed in Sections 2.7.2.2 and 2.7.2.3.

NOTE: All samples should be examined microscopically before and after each matrix reduction step to monitor changes in sample particle size, composition, and crystallinity, and to ensure sample representativeness and homogeneity for analysis.

2.7.2.1.1 *Milling*—Mechanical milling of asbestos materials has been shown to decrease fiber crystallinity, with a resultant decrease in diffraction intensity of the specimen; the degree of lattice alteration is related to the dura-

tion and type of milling process.^{19,22} Therefore, all milling times should be kept to a minimum.

For *qualitative analysis*, particle size is not usually of critical importance and initial characterization of the material with a minimum of matrix reduction is often desirable to document the composition of the sample as received. Bulk samples of very large particle size (>2–3 mm) should be comminuted to ~100 µm. A mortar and pestle can sometimes be used in size reduction of soft or loosely bound materials though this may cause matting of some samples. Such samples may be reduced by cutting with a razor blade in a mortar, or by grinding in a suitable mill (e.g., a microhammer mill or equivalent). When using a mortar for grinding or cutting, the sample should be moistened with ethanol, or some other suitable wetting agent, to minimize exposures.

For accurate, reproducible *quantitative analysis*, the particle size of both sample and standard materials should be reduced to ~10 µm (see Section 2.3.3). Dry ball milling at liquid nitrogen temperatures (e.g., Spex Freezer Mill, or equivalent) for a maximum time of 10 min. is recommended to obtain satisfactory particle size distributions while protecting the integrity of the crystal lattice.⁵ Bulk samples of very large particle size may require grinding in two stages for full matrix reduction to <10 µm.^{8, 16}

Final particle size distributions should always be verified by optical microscopy or another suitable method.

2.7.2.1.2 Low temperature ashing—For materials shown by PLM to contain large amounts of gypsum, cellulosic, or other organic materials, it may be desirable to ash the samples prior to analysis to reduce background radiation or matrix interference. Since chrysotile undergoes dehydroxylation at temperatures between 550° C and 650° C, with subsequent transformation to forsterite,^{23, 24} ashing temperatures should be kept below 500° C. Use of a low temperature asher is recommended. In all cases, calibration of the oven is essential to ensure that a maximum ashing temperature of 500° C is not exceeded.

2.7.2.1.3 Acid leaching—Because of the interference caused by gypsum and some carbonates in the detection of asbestiform minerals by XRD (see Section 2.3.1), it may be necessary to remove these interferents by a simple acid leaching procedure prior to analysis (see Section 1.7.2.2).

2.7.2.2 Qualitative Analysis

2.7.2.2.1 Initial screening of bulk material—Qualitative analysis should be performed on a representative, homogeneous portion of the sample with a minimum of sample treatment.

1. Grind and mix the sample with a mortar and pestle (or equivalent method, see Section 2.7.2.1.1.) to a final particle size sufficiently small (~100 µm) to allow adequate packing into the sample holder.

2. Pack the sample into a standard bulk sample holder. Care should be taken to ensure that a representative portion of the milled sample is selected for analysis. Particular care should be taken to avoid possible size segregation of the sample. (Note: Use of a back-packing method²⁵ of bulk sample preparation may reduce preferred orientation effects.)

3. Mount the sample on the diffractometer and scan over the diagnostic peak regions for the serpentine (~6.7 Å) and amphibole (8.2–8.5 Å) minerals (see Table 2–2). The X-ray diffraction equipment should be optimized for intensity. A slow scanning speed of 1° 2θ/min is recommended for adequate resolution. Use of a sample spinner is recommended.

ommended for adequate resolution. Use of a sample spinner is recommended.

4. Submit all samples that exhibit diffraction peaks in the diagnostic regions for asbestiform minerals to a full qualitative XRD scan (5°–60° 2θ; 1°2θ/min) to verify initial peak assignments and to identify potential matrix interferences when subsequent quantitative analysis is to be performed.

5. Compare the sample XRD pattern with standard reference powder diffraction patterns (i.e., JCPDS powder diffraction data³ or those of other well-characterized reference materials). Principal lattice spacings of asbestiform minerals are given in Table 2–2; common constituents of bulk insulation and wall materials are listed in Table 2–3.

2.7.2.2.2 Detection of minor or trace constituents—Routine screening of bulk materials by XRD may fail to detect small concentrations (<5 percent) of asbestos. The limits of detection will, in general, be improved if matrix absorption effects are minimized, and if the sample particle size is reduced to the optimal 1 to 10 µm range, provided that the crystal lattice is not degraded in the milling process. Therefore, in those instances where confirmation of the presence of an asbestiform mineral at very low levels is required, or where a negative result from initial screening of the bulk material by XRD (see Section 2.7.2.2.1) is in conflict with previous PLM results, it may be desirable to prepare the sample as described for quantitative analysis (see Section 2.7.2.3) and step-scan over appropriate 2θ ranges of selected diagnostic peaks (Table 2–2). Accurate transfer of the sample to the silver membrane filter is not necessary unless subsequent quantitative analysis is to be performed.

2.7.2.3 Quantitative Analysis

The proposed method for quantitation of asbestos in bulk samples is a modification of the NIOSH-recommended thin-layer method for chrysotile in air.⁵ A thick-layer or bulk method involving pelletizing the sample may be used for semiquantitative analysis;^{7,8} however, this method requires the addition of an internal standard, use of a specially fabricated sample press, and relatively large amounts of standard reference materials. Additional research is required to evaluate the comparability of thin- and thick-layer methods for quantitative asbestos analysis.

For quantitative analysis by thin-layer methods, the following procedure is recommended:

1. Mill and size all or a substantial representative portion of the sample as outlined in Section 2.7.2.1.1.
2. Dry at 100° C for 2 hr; cool in a desiccator.
3. Weigh accurately to the nearest 0.01 mg.
4. Samples shown by PLM to contain large amounts of cellulosic or other organic materials, gypsum, or carbonates, should be submitted to appropriate matrix reduction procedures described in Sections 2.7.2.1.2 and 2.7.2.1.3. After ashing and/or acid treatment, repeat the drying and weighing procedures described above, and determine the percent weight loss; L.
5. Quantitatively transfer an accurately weighed amount (50–100 mg) of the sample to a 1-L volumetric flask with approximately 200 mL isopropanol to which 3 to 4 drops of surfactant have been added.
6. Ultrasonicate for 10 min at a power density of approximately 0.1 W/mL, to disperse the sample material.
7. Dilute to volume with isopropanol.
8. Place flask on a magnetic stirring plate. Stir.

9. Place a silver membrane filter on the filtration apparatus, apply a vacuum, and attach the reservoir. Release the vacuum and add several milliliters of isopropanol to the reservoir. Vigorously hand shake the asbestos suspension and immediately withdraw an aliquot from the center of the suspension so that total sample weight, W_T , on the filter will be approximately 1 mg. Do not adjust the volume in the pipet by expelling part of the suspension; if more than the desired aliquot is withdrawn, discard the aliquot and resume the procedure with a clean pipet. Transfer the aliquot to the reservoir. Filter rapidly under vacuum. Do not wash the reservoir walls. Leave the filter apparatus under vacuum until dry. Remove the reservoir, release the vacuum, and remove the filter with forceps. (Note: Water-soluble matrix interferences such as gypsum may be removed at this time by careful washing of the filtrate with distilled water. Extreme care should be taken not to disturb the sample.)

10. Attach the filter to a flat holder with a suitable adhesive and place on the diffractometer. Use of a sample spinner is recommended.

11. For each asbestos mineral to be quantitated select a reflection (or reflections) that has been shown to be free from interferences by prior PLM or qualitative XRD analysis and that can be used unambiguously as an index of the amount of material present in the sample (see Table 2-2).

12. Analyze the selected diagnostic reflection(s) by step scanning in increments of $0.02^\circ 2\theta$ for an appropriate fixed time and integrating the counts. (A fixed count scan may be used alternatively; however, the method chosen should be used consistently for all samples and standards.) An appropriate scanning interval should be selected for each peak, and background corrections made. For a fixed time scan, measure the background on each side of the peak for one-half the peak-scanning time. The net intensity, I_a , is the difference between the peak integrated count and the total background count.

13. Determine the net count, I_{Ag} , of the filter 2.36 Å silver peak following the procedure in step 12. Remove the filter from the holder, reverse it, and reattach it to the holder. Determine the net count for the unattenuated silver peak, I_{Ag} . Scan times may be less for measurement of silver peaks than for sample peaks; however, they should be constant throughout the analysis.

14. Normalize all raw, net intensities (to correct for instrument instabilities) by referencing them to an external standard (e.g., the 3.34 Å peak of an α -quartz reference crystal). After each unknown is scanned, determine the net count, I , of the reference specimen following the procedure in step 12. Determine the normalized intensities by dividing the peak intensities by I :

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2.8 Calibration

2.8.1 Preparation of Calibration Standards

1. Mill and size standard asbestos materials according to the procedure outlined in Section 2.7.2.1.1. *Equivalent, standardized matrix reduction and sizing techniques should be used for both standard and sample materials.*

2. Dry at 100°C for 2 hr; cool in a desiccator.

3. Prepare two suspensions of each standard in isopropanol by weighing approximately 10 and 50 mg of the dry material to the nearest 0.01 mg. Quantitatively transfer each to a 1-L volumetric flask with approxi-

mately 200 mL isopropanol to which a few drops of surfactant have been added.

4. Ultrasonicate for 10 min at a power density of approximately 0.1 W/mL, to disperse the asbestos material.

5. Dilute to volume with isopropanol.

6. Place the flask on a magnetic stirring plate. Stir.

7. Prepare, in triplicate, a series of at least five standard filters to cover the desired analytical range, using appropriate aliquots of the 10 and 50 mg/L suspensions and the following procedure.

Mount a silver membrane filter on the filtration apparatus. Place a few milliliters of isopropanol in the reservoir. Vigorously hand shake the asbestos suspension and immediately withdraw an aliquot from the center of the suspension. Do not adjust the volume in the pipet by expelling part of the suspension; if more than the desired aliquot is withdrawn, discard the aliquot and resume the procedure with a clean pipet. Transfer the aliquot to the reservoir. Keep the tip of the pipet near the surface of the isopropanol. Filter rapidly under vacuum. Do not wash the sides of the reservoir. Leave the vacuum on for a time sufficient to dry the filter. Release the vacuum and remove the filter with forceps.

2.8.2 Analysis of Calibration Standards

1. Mount each filter on a flat holder. Perform step scans on selected diagnostic reflections of the standards and reference specimen using the procedure outlined in Section 2.7.2.3, step 12, and the same conditions as those used for the samples.

2. Determine the normalized intensity for each peak measured, I_{std} , as outlined in Section 2.7.2.3, step 14.

2.9 Calculations

For each asbestos reference material, calculate the exact weight deposited on each standard filter from the concentrations of the standard suspensions and aliquot volumes. Record the weight, w , of each standard. Prepare a calibration curve by regressing I_{std} on w . Poor reproducibility (± 15 percent RSD) at any given level indicates problems in the sample preparation technique, and a need for new standards. The data should fit a straight line equation.

Determine the slope, m , of the calibration curve in counts/microgram. The intercept, b , of the line with the I_{std} axis should be approximately zero. A large negative intercept indicates an error in determining the background. This may arise from incorrectly measuring the baseline or from interference by another phase at the angle of background measurement. A large positive intercept indicates an error in determining the baseline or that an impurity is included in the measured peak.

Using the normalized intensity, I_{Ag} , for the attenuated silver peak of a sample, and the corresponding normalized intensity from the unattenuated silver peak, I_{Ag} , of the sample filter, calculate the transmittance, T , for each sample as follows:^{26 27}

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Determine the correction factor, $f(T)$, for each sample according to the formula:

$$f(T) = \frac{-R(\ln T)}{1 - T^R}$$

where

§ 763.99

$$R = \frac{\sin \Theta_{Ag}}{\sin \Theta_s}$$

Θ_{Ag} =angular position of the measured silver peak (from Bragg's Law), and

Θ_s =angular position of the diagnostic asbestos peak.

Calculate the weight, W_s , in micrograms, of the asbestos material analyzed for in each sample, using the appropriate calibration data and absorption corrections: EC01AP92.020

Calculate the percent composition, P_s , of each asbestos mineral analyzed for in the parent material, from the total sample weight, W_T , on the filter:

$$P_s = \frac{W_s(1-0.01L)}{W_T} \times 100$$

where

P_s =percent asbestos mineral in parent material;

W_s =mass of asbestos mineral on filter, in μg ;

W_T =total sample weight on filter, in μg ;

L =percent weight loss of parent material on ashing and/or acid treatment (see Section 2.7.2.3).

2.10 References

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[47 FR 23369, May 27, 1982; 47 FR 38535, Sept. 1, 1982; Redesignated at 60 FR 31922, June 19, 1995]

Subpart F [Reserved]

Subpart G—Asbestos Abatement Projects

SOURCE: 52 FR 5623, Feb. 25, 1987, unless otherwise noted.

§ 763.120 Scope

(a) This part establishes requirements which must be followed during asbestos abatement projects by employers of State and local government employees not covered by the Asbestos Standard of the Occupational Safety and Health Administration (OSHA), 29 CFR 1926.58, an Asbestos Standard adopted by a State as part of a State plan approved by OSHA under section 18 of the Occupational Safety and Health Act, or a State asbestos regulation which EPA has determined to be comparable to or more stringent than this part. The rule covers those employees who take part in asbestos abatement work.

(b) [Reserved]

§ 763.121 Regulatory requirements.

(a) [Reserved]

(b) *Definitions.* *Action level* means an airborne concentration of asbestos of 0.1 fiber per cubic centimeter (f/cc) of air calculated as an 8-hour time-weighted average.

Administrator means the Administrator, U.S. Environmental Protection Agency, or designee.

Asbestos means the asbestiform varieties of chrysotile (serpentine); crocidolite (riebeckite); amosite (cummingtonite—grunerite); tremolite; anthophyllite, and actinolite.

Asbestos abatement project means any activity involving the removal, enclosure, or encapsulation of friable asbestos material.

Authorized person means any person authorized by the employer and required by work duties to be present in regulated areas.

Clean room means an uncontaminated room having facilities for the storage of employees' street clothing and uncontaminated materials and equipment.

Competent person means one who is capable of identifying existing asbestos hazards in the workplace and who has the authority to take prompt corrective measures to eliminate them. The duties of the competent person include at least the following: Establishing the negative-pressure enclosure, ensuring its integrity, and controlling entry to and exit from the enclosure; supervising any em-

ployee exposure monitoring required by this subpart, ensuring that all employees working within such an enclosure wear the appropriate personal protective equipment, are trained in the use of appropriate methods of exposure control, and use the hygiene facilities and decontamination procedures specified in this subpart; and ensuring that engineering controls in use are in proper operating condition and are functioning properly.

Decontamination area means an enclosed area adjacent and connected to the regulated area and consisting of an equipment room, shower area, and clean room, which is used for the decontamination of workers, materials, and equipment contaminated with asbestos.

Demolition means the wrecking or taking out of any load-supporting structural member and any related razing, removing, or stripping of asbestos products.

Emergency project means a project involving the removal, enclosure, or encapsulation of friable asbestos-containing material that was not planned but results from a sudden unexpected event.

Employee exposure means that exposure to airborne asbestos would occur if the employee were not using respiratory protective equipment.

Employer means the public department, agency, or entity which hires an employee. The term includes, but is not limited to, any State, County, City, or other local governmental entity which operates or administers schools, a department of health or human services, a library, a police department, a fire department, or similar public service agencies or offices.

Equipment room (change room) means a contaminated room located within the decontamination area that is supplied with impermeable bags or containers for the disposal of contaminated protective clothing and equipment.

Fiber means a particulate form of asbestos, 5 micrometers or longer, with a length-to-diameter ratio of at least 3 to 1.

Friable asbestos material means any material containing more than 1 percent asbestos by weight which, when dry, may be crumbled, pulverized, or reduced to powder by hand pressure.

High-efficiency particulate air (HEPA) filter means a filter capable of trapping and retaining at least 99.97 percent of all monodispersed particles of 0.3 micrometer in diameter or larger.

Regulated area means an area established by the employer to demarcate areas where airborne concentrations of asbestos exceed or can reasonably be expected to exceed the permissible exposure limit. The regulated area may take the form of: (1) A temporary enclosure, as required by paragraph (e)(6) of this section, or (2) an area demarcated in any manner that minimizes the number of employees exposed to asbestos.



UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
REGION 5
77 WEST JACKSON BOULEVARD
CHICAGO, IL 60604-3590

REPLY TO THE ATTENTION OF:

**VIA TELEFAX AND
REGULAR MAIL.**

April 29, 2002

Denny Clinton, Project Manager
Johns Manville
1871 North Pershing Road
Waukegan, IL 60087

Dear Mr. Clinton:

The U.S. Environmental Protection Agency (EPA) and the Illinois EPA have reviewed the draft document entitled "Sediment Sampling and Analysis Plan and Conceptual Closure Schedule, Second Explanation of Significant Differences; Johns Manville, Waukegan, Illinois" and hereby approve this document with the modifications listed below in comments #2b, 3, and 4:

1. Page 1, point #2- In accordance with the Second Explanation of Significant Differences (ESD), January 1, 2004 is the latest date that the Settling Basin closure could be completed. The remaining ponds and waterways in the Johns-Manville- Waukegan, Illinois Superfund Site (the Site) must be closed as soon as possible.

2. a. Page 2, "Sediment Sampling Locations" Paragraph- it is suggested that the transects be angled, rather than oriented straight across the ponds.

b. Additionally, due to the relative size and potential for non-uniform deposition, sampling shall be conducted along three transects in the Industrial Canal. Sampling along one transect in the Borrow Pit shall also be conducted. The Borrow Pit transect should be oriented across the center of the original Borrow Pit area, not in the portions that were excavated as part of the Superfund remedy for the site. Figure 1 shall also be revised accordingly.

3. The On-Site Landfill shall include the portion of the Collection Basin which has been filled.

4. As outlined above, closure of all former wastewater treatment system ponds and waterways must be completed by January 1, 2004. The conceptual schedule for closure of the former wastewater treatment ponds is amended to indicate that January 1, 2004 is the "finish" date for item #18, "Construction".

Please begin the implementation of this conceptual closure work plan by submitting the "Sample

Recycled/Recyclable . Printed with Vegetable Oil Based Inks on 100% Recycled Paper 150% Postconsumer

APR 29 2002 16:13

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PAGE.03

-2-

Preparation and Analysis Protocol", which was due, per your schedule, on March 14, 2002, and the "Draft Closure Plan- Configuration", which was due on March 6, 2002.

If you have any questions concerning this letter, please contact me at (312) 886-4742.

Sincerely,



Brad Bradley
Remedial Project Manager

cc: Sandy Bron, Illinois EPA
Beth Wallace, IAG Office

APR 29 2002 16:13

312 353 5541

PAGE.03

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904 786 0213

PAGE.04

Prepared by:

Date:

5/16/02

1

2

To: Bill Bow 847-695-7799

3

4

From: Denny

5

6

No Pages: 4 (incl. cover)

7

8

Bill:

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11

LETTER WE DISCUSSED

12

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W. D. Clinton

Project Manager
1871 North Pershing Road
Waukegan, Illinois 60087
(847) 360-4490

February 14, 2002

Mr. Brad Bradley
Project Manager
United States Environmental Protection Agency Region 5
77 West Jackson Boulevard
Chicago, IL 60604-3507

COPY

Subject: Sediment Sampling and Analysis Plan and Conceptual Closure Schedule, Second Explanation of Significant Differences; Johns Manville, Waukegan, Illinois

Dear Mr. Bradley:

In accordance with your request dated December 3, 2001 and correspondence from Johns Manville (JM) dated January 18, 2002, we are submitting this Sediment Sampling and Analysis Plan and Conceptual Schedule for implementation of the various requirements in the Second Explanation of Significant Differences (ESD) issued for the JM property in Waukegan, Illinois (the "Site"). This proposed plan is also in response to ongoing communication between JM and U.S. EPA regarding final closure at Site.

Dated September 22, 2000, the Second ESD includes, but is not limited to, the following general provisions:

1. The sediments within the Collection Basin, Industrial Canal, and Pumping Lagoon will be sampled for the presence of asbestos. Final closure requirements for these three basins will be established following receipt of the sample results.
2. The Paper Mill Ditch, Catch Basin, Mixing Basin Stages 3, 4, and 5, and Settling Basin shall be closed by January 1, 2004 in accordance with the cover requirements in the 1987 Record of Decision.
3. The On-Site Landfill shall be closed in accordance with applicable Illinois requirements.

As per Item #1 (above), this correspondence contains a Sediment Sampling and Analysis Plan for determining the presence of asbestos in the sediments within the Collection Basin, Industrial Canal, and Pumping Lagoon. As per Items #2 and #3 (above), attached is a Conceptual Closure Schedule.

Sediment Sampling and Analysis Plan

This Sediment Sampling and Analysis Plan (SAP) is designed to determine whether asbestos-containing materials and/or fibers are present within the sediments of the Collection Basin, Industrial Canal, and Pumping Lagoon.

Sediment Sample Locations

As you requested, sediment samples will be collected along five separate transect lines across the Pumping Lagoon (1 transect), Industrial Canal (2 transects), and Collection Basin (2 transects). Three sample locations will be established for each transect; one each on opposing shorelines intersecting the transect line and one located at the midpoint of the transect line. See Figure 1 for a visual representation of the sample locations.

The shoreline sediment samples will be collected within twenty-five feet of the shoreline, below the current water level. The midpoint sample will be collected at the approximate halfway location between each shoreline along each transect.

Using this methodology, three sediment samples will be collected from the Pumping Lagoon, six sediment samples from the Industrial Canal, and six sediment samples from the Collection Basin. The samples from each of the fifteen sample locations will be analyzed separately; no sample compositing will be done between sample locations.

Sediment Sample Collection

The sediment samples will be collected using an AMS 3.25-inch x 12-inch stainless-steel sludge sampler. This sampler contains a butterfly valve at its base, thereby preventing sediments from escaping from the sampler after penetrating the water-sediment interface. Each sample will be collected within a new 3-inch x 12-inch Butyrate (plastic) liner placed within the sampler. The sample will be collected by inserting the sampler into the upper twelve inches of the sediment column and then withdrawing the sampler back to the water surface. After the sample has been collected and the liner removed from the sampler, end caps will be placed over the tube ends, the tube labeled, and retained for analysis. New liners will be used at each sample location; the sludge sampler will be rinsed of all visible debris prior to collecting each sediment sample.

Quality Assurance/Quality Control

Quality Assurance/Quality Control (QA/QC) shall consist of collecting one field duplicate sediment sample at one of the pre-established sample locations. The location of the field duplicate will be determined at random on the day of the sampling. The sample will be labeled as "Field Duplicate", however, its location will not be made known to the laboratory.

Sediment Sampling and Analysis Plan and Conceptual Closure Schedule
February 14, 2002
Page 3

Sediment Sample Analysis

As you requested, sediment sample analysis for asbestos shall be conducted using Polarized Light Microscopy (PLM). We are currently reviewing the suitability of the various sediment sample preparation methods available, including the Region 1 protocols developed specifically for sediment analysis. We will submit, for your approval, a sediment sample preparation and analysis protocol (using PLM) no later than March 14, 2002.

Report

All results, including QA/QC analyses, will be compiled into tables. The report will include all field notes, sample inspections, and laboratory data. Photographs of each sample location will also be recorded and included in the report.

Conceptual Closure Schedule

Attachment 2 contains the Conceptual Closure Schedule for implementing the requirements of the Second ESD, including the Sediment Sampling and Analysis Plan.

If you have any questions regarding this plan, please contact me at (303) 808-2127.

Sincerely,



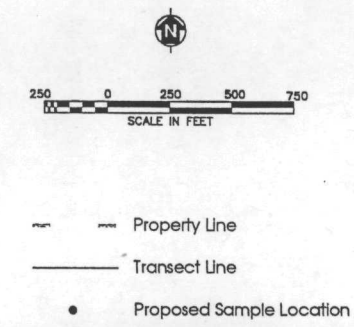
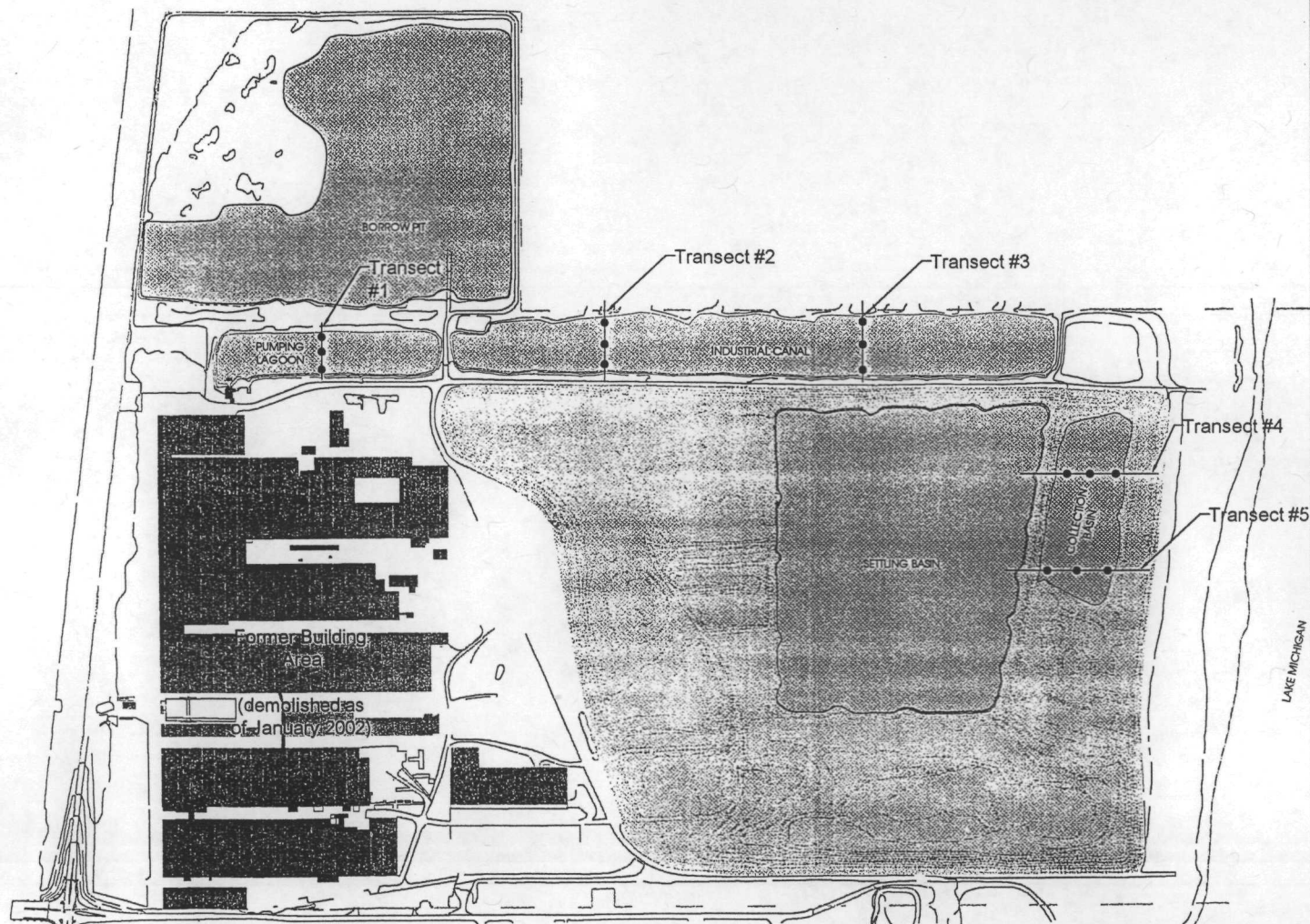
W. Dennis Clinton
Project Manager

Attachments

cc: Bruce Ray/JM
William Bow/LFR
Edward Kenney/Sidley & Austin
David Peterson/David M. Peterson, PE, PC

Attachment 1

Sediment Sample Location Map



JOHNS MANVILLE, WAUKEGAN, ILLINOIS

**Proposed Transect Lines
and Sediment
Sample Locations**



Figure 1

Attachment 2

Conceptual Closure Schedule

2nd ESD Conceptual Implementation Schedule

ID	Task Name	Duration	Start	Finish	2002				2003				2004			
					Qtr 1	Qtr 2	Qtr 3	Qtr 4	Qtr 1	Qtr 2	Qtr 3	Qtr 4	Qtr 1	Qtr 2	Qtr 3	Qtr 4
1	Collection Basin, Industrial Canal, Pumping Lagoon Closure	115 days	Fri 2/15/02	Thu 7/25/02												
2	Sediment Sampling	75 days	Fri 2/15/02	Thu 5/30/02												
3	Work Plan to USEPA	0 days	Fri 2/15/02	Fri 2/15/02												
4	Submit Sample Preparation and Analysis Protocol	4 wks	Fri 2/15/02	Thu 3/14/02												
5	US EPA Approval	4 wks	Fri 3/15/02	Thu 4/11/02												
6	Sample Collection	1 wk	Fri 4/12/02	Thu 4/18/02												
7	Laboratory Analysis	2 wks	Fri 4/19/02	Thu 5/2/02												
8	Report	4 wks	Fri 5/3/02	Thu 5/30/02												
9	Closure Plan	40 days	Fri 5/31/02	Thu 7/25/02												
10	Depends upon sediment samples	8 wks	Fri 5/31/02	Thu 7/25/02												
11	Settling Basin et al Closure	750 days	Thu 2/7/02	Wed 12/22/04												
12	Draft Closure Plan-Configuration	4 wks	Thu 2/7/02	Wed 3/6/02												
13	Feasibility Study	12 wks	Thu 3/7/02	Wed 5/29/02												
14	Pilot Test	4 wks	Thu 5/30/02	Wed 6/26/02												
15	Final Closure Plan	12 wks	Thu 6/27/02	Wed 9/18/02												
16	Design	8 wks	Thu 9/19/02	Wed 11/13/02												
17	Bid	6 wks	Thu 11/14/02	Wed 12/25/02												
18	Construction	104 wks	Thu 12/26/02	Wed 12/22/04												

Project: Schedule-07Feb02
Date: Thu 2/14/02

Task

Split

Progress

Milestone

Summary

Rolled Up Task

Rolled Up Split

Rolled Up Milestone

Rolled Up Progress

External Tasks

Project Summary

2nd ESD Conceptual Implementation Schedule

ID	Task Name	Duration	Start	Finish	2002				2003				2004			
					Qtr 1	Qtr 2	Qtr 3	Qtr 4	Qtr 1	Qtr 2	Qtr 3	Qtr 4	Qtr 1	Qtr 2	Qtr 3	Qtr 4
19	On-Site Landfill Closure	612 days	Thu 2/7/02	Fri 6/11/04												
20	Leachate Investigation	141 days	Thu 2/7/02	Thu 8/22/02												
21	Work Plan	8 wks	Thu 2/7/02	Wed 4/3/02												
22	Meet with IEPA	1 day	Thu 4/4/02	Thu 4/4/02												
23	Finalize Plan and Submit for IEPA Review	6 wks	Fri 4/5/02	Thu 5/16/02												
24	Monitoring Well Installation	4 wks	Fri 5/17/02	Thu 6/13/02												
25	Site Survey	4 wks	Fri 6/14/02	Thu 7/11/02												
26	Leachate Sample Collection	1 wk	Fri 6/14/02	Thu 6/20/02												
27	Laboratory Analysis	3 wks	Fri 6/21/02	Thu 7/11/02												
28	Report Development	6 wks	Fri 7/12/02	Thu 8/22/02												
29	Develop and Submit Closure Plan	61 days	Fri 8/23/02	Fri 11/15/02												
30	Develop Draft	4 wks	Fri 8/23/02	Thu 9/19/02												
31	Meet w/ IEPA	1 day	Fri 9/20/02	Fri 9/20/02												
32	Finalize Plan	4 wks	Mon 9/23/02	Fri 10/18/02												
33	Finalize Plan and Submit for IEPA Review	4 wks	Mon 10/21/02	Fri 11/15/02												
34	Adjusted Standard Process	150 days	Mon 11/18/02	Fri 6/13/03												
35	Develop and Submit Adjusted Standard Petition	4 wks	Mon 11/18/02	Fri 12/13/02												
36	Adjusted Standard Review and Approval Process	26 wks	Mon 12/16/02	Fri 6/13/03												
37	Implement Closure Plan	52 wks	Mon 6/16/03	Fri 6/11/04												

Project: Schedule-07Feb02
Date: Thu 2/14/02

Task

Split

Progress

Milestone

Summary

Rolled Up Task

Rolled Up Split

Rolled Up Milestone

Rolled Up Progress

External Tasks

Project Summary

Appendix B

Sediment Sample Analytical Report

EMSL Analytical, Inc.

107 Haddon Ave., Westmont, NJ 08108

Phone: (856) 858-4800 Fax: (856) 858-4960 Email: ssiegel@EMSL.com**EMSL**Attn: Wendy Teskey
LFR Levine Friche
630 Tollgate Rd.
Suite D
Elgin, IL 60123Fax: (847) 695-7799
Project: JM-009-07992-00-001

Phone: 847-695-8855

Customer ID: LEVI93
Customer PO:
Received: 06/20/02 12:33 PM

EMSL Order: 040208906

EMSL Project ID:


Analysis Date: 6/27/2002

EPA Protocol for Screening Soil and Sediment Samples for Asbestos Content Used by USEPA, Region 1 Laboratory (Rev May 24, 1994). Modified by EMSL (Sept 1999)

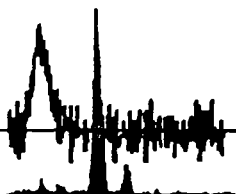
Sample	Location	% Asbestos
SS-PL-1A 040208906-0001		2.4 Amosite 4.4 Chrysotile
SS-PL-1B 040208906-0002		2.2 Amosite 2.5 Chrysotile
SS-PL-1C 040208906-0003		<0.25% Chrysotile
SS-IC-2A 040208906-0004		3.4 Chrysotile
SS-IC-2B 040208906-0005		1.7 Amosite 3.9 Chrysotile
SS-IC-2C 040208906-0006		1.7 Amosite 4.0 Chrysotile
SS-IC-3A 040208906-0007		3.5 Chrysotile
SS-IC-3B 040208906-0008		1.1 Amosite 3.3 Chrysotile

Analyst(s)

Delores Beard (22)


Stephen Siegel, CIH
or other approved signatory

PLM-1



EMSL Analytical, Inc.

107 Haddon Ave., Westmont, NJ 08108

Phone: (856) 858-4800 Fax: (856) 858-4960 Email: ssiegel@EMSL.com**EMSL**Attn: Wendy Teskey
LFR Levine Friche
630 Tollgate Rd.
Suite D
Elgin, IL 60123Fax: (847) 695-7799
Project: JM-009-07992-00-001

Phone: 847-695-8855

Customer ID: LEVI93
Customer PO:
Received: 06/20/02 12:33 PMEMSL Order: 040208906
EMSL Project ID:
Analysis Date: 6/27/2002**EPA Protocol for Screening Soil and Sediment Samples for Asbestos Content Used by USEPA, Region 1 Laboratory (Rev May 24, 1994). Modified by EMSL (Sept 1999)**

Sample	Location	% Asbestos
SS-IC-3C		1.0 Chrysotile
040208906-0009		

SS-IC-4A		3.2 Chrysotile
040208906-0010		

SS-IC-4B		1.0 Chrysotile
040208906-0011		

SS-IC-4C		1.9 Chrysotile
040208906-0012		

SS-CB-5A		1.9 Chrysotile
040208906-0013		
		<0.25% Crocidolite

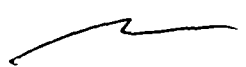
SS-CB-5B		1.0 Amosite
040208906-0014		
		2.0 Chrysotile

SS-CB-5C		1.4 Chrysotile
040208906-0015		
		<1.0% Crocidolite

SS-CB-6A		1.1 Chrysotile
040208906-0016		

Analyst(s)

Delores Beard (22)


Stephen Siegel, CIH
or other approved signatory

PLM-1



EMSL Analytical, Inc.

107 Haddon Ave., Westmont, NJ 08108

Phone: (856) 858-4800 Fax: (856) 858-4960 Email: ssiege@EMSL.com**EMSL**Attn: Wendy Teskey
LFR Levine Friche
630 Tollgate Rd.
Suite D
Elgin, IL 60123Fax: (847) 695-7799
Project: JM-009-07992-00-001

Phone: 847-695-8855

Customer ID: LEVI93
Customer PO:
Received: 06/20/02 12:33 PMEMSL Order: 040208906
EMSL Project ID:
Analysis Date: 6/27/2002**EPA Protocol for Screening Soil and Sediment Samples for Asbestos Content Used by USEPA, Region 1 Laboratory (Rev May 24, 1994). Modified by EMSL (Sept 1999)**

Sample	Location	% Asbestos
SS-CB-6B 040208906-0017		None Detected

SS-CB-6C 040208906-0018		0.4 Chrysotile
----------------------------	--	----------------

SS-BP-7A 040208906-0019		<0.25% Chrysotile
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SS-BP-7B 040208906-0020		<0.25% Chrysotile
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SS-BP-7C 040208906-0021		None Detected
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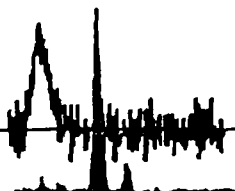
FIELD DUPLICATE 040208906-0022		None Detected
-----------------------------------	--	---------------

Analyst(s)

Delores Beard (22)


Stephen Siegel, CIH
or other approved signatory

PLM-1





CHAIN OF CUSTODY

Revised January 1, 2000

EMSL Rep: Paul NyfieldThird Party Billing requires written authorization
from third partyYour Company Name: LFR Levine-FrickeEMSL-Bill to: SameStreet: 630 Tollgate Rd.

Street: _____

Box #: Suite D

Box #: _____

City/State: Elgin, IL Zip: 60123

City/State: _____

Zip: _____

Phone Results to:

Fax Results to:

Name: Wendy TeskeyName: SameTelephone #: 847-695-8855 ext. 112Fax #: 847-695-7799

Project

Purchase Order #:

Name/Number: JM-009-07992-00-001

MATRIX

TURNAROUND

<input type="checkbox"/> Air	<input checked="" type="checkbox"/> Soil	<input type="checkbox"/> Micro-Vac	<input type="checkbox"/> 3 hrs	<input type="checkbox"/> 6 Hours	<input type="checkbox"/> Same Day or 12 Hours*	<input type="checkbox"/> 24 Hours (1 day)
<input type="checkbox"/> Bulk	<input type="checkbox"/> Drinking Water		<input type="checkbox"/> 48 Hours (2 days)	<input type="checkbox"/> 72 Hours (3 days)	<input type="checkbox"/> 96 Hours (4 days)	<input type="checkbox"/> 120 Hours (5 Days)
<input type="checkbox"/> Wipe	<input type="checkbox"/> Wastewater		<input checked="" type="checkbox"/> 144+ hours (6-10 Days)			

TEM AIR, 3 hours, 6 hours, Please call ahead to schedule. There is a premium charge for 3-hour tat; please call 1-800-220-3675 for price prior to sending samples. You will be asked to sign an authorization form for this service.

*12 hours (must arrive by 11:00 a.m Mon - Fri.), Please Refer to Price Quote

PCM - Air <input type="checkbox"/> NIOSH 7400(A) Issue 2: August 1994 <input type="checkbox"/> OSHA w/ TWA <input type="checkbox"/> Other:	TEM Air <input type="checkbox"/> AHERA 40 CFR, Part 763 Subpart E <input type="checkbox"/> NIOSH 7402 Issue 2 <input type="checkbox"/> EPA Level II	TEM Water <input type="checkbox"/> EPA 100.1 <input type="checkbox"/> EPA 100.2 <input type="checkbox"/> NYS 198.2
PLM - Bulk <input type="checkbox"/> EPA 600/R-93/116 <input type="checkbox"/> EPA Point Count <input type="checkbox"/> NYS Stratified Point Count <input type="checkbox"/> PLM NOB (Gravimetric) NYS 198.1 <input type="checkbox"/> NIOSH 9002 <input type="checkbox"/> EMSL Standard Addition	TEM Bulk <input type="checkbox"/> Drop Mount (Qualitative) <input type="checkbox"/> Chatfield SOP - 1988-02 <input type="checkbox"/> TEM NOB (Gravimetric) NYS 198.4 <input type="checkbox"/> EMSL Standard Addition	TEM Microvan/Wipe <input type="checkbox"/> ASTM D 2755-95 quantitative <input type="checkbox"/> Wipe Qualitative
SEM Air or Bulk <input type="checkbox"/> Qualitative <input type="checkbox"/> Quantitative	PLM Soil <input type="checkbox"/> EPA Protocol Qualitative <input checked="" type="checkbox"/> EPA Protocol Quantitative (90 asbestos) <input type="checkbox"/> EMSL MSD 9000 Method fibers/gram	XRD <input type="checkbox"/> Asbestos <input type="checkbox"/> Silica NIOSH 7500
		OTHER <input type="checkbox"/>

Client Sample # (s) _____ Total Samples #: 22 TotalRelinquished: Wendy Teskey Date: 6/19/02 Time: 1300Received: A Kemp Date: _____ Time: _____

Relinquished: _____ Date: _____ Time: _____



CHAIN OF CUSTODY

Revised January 1, 2000

EMSL Rep: Paul Nyfield

Third Party Billing requires written authorization from third party

Your Company Name: LFR Levine-Fricke

EMSL-Bill to:

Street: 630 Tollgate Rd.

Street:

Box #: Suite D

Box #:

City/State: Elgin, IL Zip: 60123

City/State:

Zip:

Phone Results to:

Fax Results to:

Name: Wendy Teskey

Name:

Telephone #: 847-695-8855 ext 112

Fax #:

Project

Purchase Order #:

Name/Number: Jm-009-07992-00-01

MATRIX

TURNAROUND

<input type="checkbox"/> Air	<input checked="" type="checkbox"/> Soil	<input type="checkbox"/> Micro-Vac	<input type="checkbox"/> 3 hrs	<input type="checkbox"/> 6 Hours	<input type="checkbox"/> Same Day or 12 Hours*	<input type="checkbox"/> 24 Hours (1 day)
<input type="checkbox"/> Bulk	<input type="checkbox"/> Drinking Water		<input type="checkbox"/> 48 Hours (2 days)	<input type="checkbox"/> 72 Hours (3 days)	<input type="checkbox"/> 96 Hours (4 days)	<input type="checkbox"/> 120 Hours (5 Days)
<input type="checkbox"/> Wipe	<input type="checkbox"/> Wastewater		<input checked="" type="checkbox"/> 144+ hours (6-10 Days)			

TEM AIR, 3 hours, 6 hours, Please call ahead to schedule. There is a premium charge for 3-hour tat; please call 1-800-220-3675 for price prior to sending samples. You will be asked to sign an authorization form for this service.

*12 hours (must arrive by 11:00 a.m Mon - Fri.), Please Refer to Price Quote

PCM - Air

- ☐ NIOSH 7400(A) Issue 2: August 1994
☐ OSHA w/ TWA
☐ Other:

TEM Air

- ☐ AHERA 40 CFR, Part 763 Subpart E
☐ NIOSH 7402 Issue 2
☐ EPA Level II

TEM Water

- ☐ EPA 100.1
☐ EPA 100.2
☐ NYS 198.2

PLM - Bulk

- ☐ EPA 600/R-93/116
☐ EPA Point Count
☐ NYS Stratified Point Count
☐ PLM NOB (Gravimetric) NYS 198.1
☐ NIOSH 9002
☐ EMSL Standard Addition

TEM Bulk

- ☐ Drop Mount (Qualitative)
☐ Chatfield SOP - 1988-02
☐ TEM NOB (Gravimetric) NYS 198.4
☐ EMSL Standard Addition

TEM Microvac/Wipe

- ☐ ASTM D 5755-95 quantitative
☐ Wipe Qualitative

SEM Air or Bulk

- ☐ Qualitative
☐ Quantitative

PLM Soil

- ☐ EPA Protocol Qualitative
☒ EPA Protocol Quantitative (8 Asbestos)
☐ EMSL MSD 9000 Method fibers/gram

XRD

- ☐ Asbestos
☐ Silica NIOSH 7500

OTHER

☐

Client Sample # (s)

Total Samples #:

22 Total

Relinquished:

Date:

6/19/02

Time:

1300

Received:

Date:

Time:

Relinquished:

Date:

Time:



CHAIN OF CUSTODY

Revised January 1, 2000

EMSL Rep: Paul Nyfield

Third Party Billing requires written authorization from third party

Your Company Name: LFR Levine-Fricke

EMSL-Bill to:

Street: 630 Tollgate Rd.

Street:

Box #: Suite D

Box #:

City/State: Elgin, IL Zip: 60123

City/State:

Zip:

Phone Results to:

Name: Wendy Taskay

Fax Results to:

Telephone #: 847-695-8855 ext 112

Name:

Project

Fax #:

Name/Number: Jm-009-07992-00-01

Purchase Order #:

MATRIX

TURNAROUND

<input type="checkbox"/> Air	<input checked="" type="checkbox"/> Soil	<input type="checkbox"/> Micro-Vac	<input type="checkbox"/> 3 hrs	<input type="checkbox"/> 6 Hours	<input type="checkbox"/> Same Day or 12 Hours*	<input type="checkbox"/> 24 Hours (1 day)
<input type="checkbox"/> Bulk	<input type="checkbox"/> Drinking Water		<input type="checkbox"/> 48 Hours (2 days)	<input type="checkbox"/> 72 Hours (3 days)	<input type="checkbox"/> 96 Hours (4 days)	<input type="checkbox"/> 120 Hours (5 days)
<input type="checkbox"/> Wipe	<input type="checkbox"/> Wastewater		<input checked="" type="checkbox"/> 144+ hours (6-10 Days)			

TEM AIR, 3 hours, 6 hours, Please call ahead to schedule. There is a premium charge for 3-hour tat; please call 1-800-220-3675 for price prior to sending samples. You will be asked to sign an authorization form for this service.

*12 hours (must arrive by 11:00 a.m Mon - Fri.), Please Refer to Price Quote

PCM - Air

- ☐ NIOSH 7400(A) Issue 2: August 1994
☐ OSHA w/ TWA
☐ Other:

TEM Air

- ☐ AHERA 40 CFR, Part 763 Subpart E
☐ NIOSH 7402 Issue 2
☐ EPA Level II

TEM Water

- ☐ EPA 100.1
☐ EPA 100.2
☐ NYS 198.2

PLM - Bulk

- ☐ EPA 600/R-93/116
☐ EPA Point Count
☐ NYS Stratified Point Count
☐ PLM NOB (Gravimetric) NYS 198.1
☐ NIOSH 9002
☐ EMSL Standard Addition

TEM Bulk

- ☐ Drop Mount (Qualitative)
☐ Chatfield SOP - 1988-02
☐ TEM NOB (Gravimetric) NYS 198.4
☐ EMSL Standard Addition

TEM Microvac/Wipe

- ☐ ASTM D 5755-95 quantitative
☐ Wipe Qualitative

SEM Air or Bulk

- ☐ Qualitative
☐ Quantitative

PLM Soil

- ☐ EPA Protocol Qualitative
☒ EPA Protocol Quantitative (8 Asbestos)
☐ EMSL MSD 9000 Method fibers/gram

XRD

- ☐ Asbestos
☐ Silica NIOSH 7500

OTHER

Client Sample # (s)

Total Samples #:

22 Total

Relinquished:

Date:

Time:

Received:

Date:

Time:

Relinquished:

Date:

Time:

Appendix C

Borrow Pit Water Sample Analytical Report

TEM, Incorporated

NIST-NVLAP TEM Air Samples 101130
NIOSH-PAT PCM Fiber Count 010238
AIHA-AAR PCM Analyst 010238

443 Duane Street, Glen Ellyn, Illinois 60137 Phone (630) 790-0880 Fax (630) 790-0882

April 28, 2000

William A. Bow
CADDIS, Inc.
212 Casse Circle
St. Charles, IL 60174

Re: Asbestos in Potable Water
TEM Project 36049

Dear William:

The attached laboratory report summarizes the results of the transmission electron microscopic analyses of the water samples submitted by your company. The samples were analyzed following procedures contained in the EPA's "Analytical Method for Determination of Asbestos Fibers in Water" by Eric J. Chatfield et. al. (EPA #100.1 600/4-83-043).

Please note that to achieve an analytical sensitivity of less than or equal to 0.01 million fibers per liter (MFL) it is necessary to filter 100mL of the original sample through a polycarbonate filter. This often results in filters that are overloaded with particulate material. Therefore, smaller volumes of filtered water are required to obtain a "readable" sample.

Whenever possible our laboratory filtered 100mL's of the submitted sample. When 100mL resulted in overloaded filter samples, TEM, Inc. was obliged to filter 50mL, 25mL, 5mL or even 1mL of the submitted sample. Obviously the reduction in filterable water reduces the analytical sensitivity (limit of detection).

This report applies only to samples tested.

Sincerely,
TEM, Incorporated



Karen Buehler
Laboratory Manager

TEM, Incorporated433 Duane Street
Glen Ellyn, IL 60137Phone (630) 790-0880
Fax (630) 790-0882LABORATORY DATA SHEET FOR
WATER SAMPLES BY TEM

CLIENT CADDIS, Inc.	TEM PROJECT #36049	DATE 4/13/00
CONTACT William Bow	TURN AROUND TIME REQUIRED: 5 Working Days	
ADDRESS 212 Casse Circle	COMMENTS Johns Manville	
CITY/STATE/ZIP St. Charles, IL 60174	Detection limit for Fiber length: $\geq 10\mu\text{m}$ $\geq 0.5\mu\text{m}$ $> 0.2\mu\text{m}$	
PHONE 630-587-5306	FAX	NVLAP ID # 1130

ASBESTOS FIBERS IN WATER

CLIENT ID	TEM NO.	CLIENT DESCRIPTION	SAMPLE TEMP. °C	SAMPLE VOLUME FILTERED	ASBESTOS STRUCTURES COUNTED	ASBESTOS STRUCTURES S/MM2	ANALYTICAL SENSITIVITY MFL	ASBESTOS CONC. MFL	ANALYST INITIALS
BP1-W5	137029		3.6	10ml	0	<5.0	0.1	<0.1	KB
BP2-W5	137030		3.7	10ml	0	<5.0	0.1	<0.1	KB
BP3-W5	137031		3.4	10ml	0	<5.0	0.1	<0.1	KB
BP4-W5	137032		3.5	10ml	0	<5.0	0.1	<0.1	KB
PB5-W5	137033		3.5	10ml	0	<5.0	0.1	<0.1	KB
BP6-W5	137034		2.2	10ml	0	5.0	0.1	0.1	KB

Note: The state of Illinois Environmental Protection Agency (IEPA) as part of the USA EPA Region 5, has required analytical sensitivity for "Analytical Method for Determination of Asbestos Fibers in Water," (U.S. EPA-600/4-83-043, September, 1983 to be 100,000 fibers per liter (0.1 MFL).

Relinquished by	Date/Time	Received by	Date/Time
Logged in by	Date/Time	Analyzed by <i>Karen Buckler</i>	Date/Time <i>4-28-2000</i>

DATE: April 19, 2000

CLIENT: JOHNS MANVILLE CORP.
1871 N. Pershing Rd.
Waukegan, IL 60087

ATTENTION: GLENN BAUMAN

REFERENCE: Project ID: Caddis, Inc.

REPORT NO: 64948

DATE COLLECTED: 4-12-00
SAMPLER: JOHN CHITWOOD / CADDIS

DATE RECEIVED: 4-13-00 1005am DATE ANALYZED: 4-18-00

SUBJECT: ANALYSIS OF WATER SAMPLES BY TRANSMISSION ELECTRON
MICROSCOPY

ACCREDITED: National Institute of Standards and Technology through NVLAP (101218)
California Department of Health Services (ELAP 1119)

Samples were identified as:

BP1-W5	WATER
BP2-W5	WATER
BP3-W5	WATER
BP4-W5	WATER
BP5-W5	WATER
BP6-W5	WATER

The date and time of collection and filtration are as follows:

<u>Sample #</u>	<u>Date/Time of Collection</u>	<u>Date/Time of Filtration</u>
BP1-W5	4-12-00/0945	4-13-00/1115am
BP2-W5	4-12-00/1015	4-13-00/1130am
BP3-W5	4-12-00/1035	4-13-00/1140am
BP4-W5	4-12-00/1050	4-13-00/1150am
BP5-W5	4-12-00/1105	4-13-00/1205pm
BP6-W5	4-12-00/1130	4-13-00/1220pm

The samples were analyzed for fibers >10 μ m in length to conform with the drinking water document, EPA 600 R 94 134. This regulation calls for an MCL (maximum contaminant level) of 7 MFL and an analytical sensitivity level of ≤ 0.2 MFL.

Report No. 64948
April 19, 2000

Page 2 of 2

Due to the turbidity of the samples, the analytical sensitivity of 0.2 MFL was not reached.

The results of the analyses and the detection limits are summarized on the following pages.

Respectfully submitted,

EMS LABORATORIES, INC.



B. M. Kolk
Laboratory Director

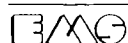
Note: The results of the analysis are based upon the samples submitted to the laboratory. No representation is made regarding the sampling area other than that implied by the analytical results for the immediate vicinity of the samples analyzed as calculated from the data presented with those samples.

This report, from a NIST accredited laboratory through NVLAP, must not be used by the client to claim product endorsement by NVLAP or any agency of the U.S. Government.

This report shall not be reproduced, except in full, without the written approval of EMS Laboratories, Inc.

Any deviation or exclusion from the test method is noted in this cover letter.

Unless otherwise noted in this cover letter, the samples were received properly packaged, clearly identified and intact.



ANALYSIS OF WATER BY TEM (EPA-600 R 94 134)

EMS NO: 64948

CLIENT: Johns Manville Tech. Center

DATE: 4/18/00

Laboratory I.D.	Client	FILTER MEDIA DATA			No. of G.O.	Analyzed Area, mm ²	Sample Volume (ml)
		Type	Diameter mm	Effective Area mm ²			
64948-1	BP 1-W 5"	PC	47	1017	10	0.092	15
64948-2	BP 2-W 5"	PC	47	1017	10	0.092	15
64948-3	BP 3-W 5"	PC	47	1017	10	0.092	15
64948-4	BP 4-W 5"	PC	47	1017	10	0.092	15
64948-5	BP 5-W 5"	PC	47	1017	10	0.092	15
64948-6	BP 6-W 5"	PC	47	1017	10	0.092	15

* FOR FIBERS > 10um ONLY

INDIVIDUAL ANALYTICAL RESULTS

Laboratory I.D.	Client I.D.	No. of Asbestos			Detection Limit (MFL)	CONCENTRATION (MFL)		
		Str	Str >5um	Str >10um		Str	Str >5um	Str >10um
64948-1	BP 1-W 5"	-	-	N.D.	0.7	-	-	N.D.
64948-2	BP 2-W 5"	-	-	N.D.	0.7	-	-	N.D.
64948-3	BP 3-W 5"	-	-	N.D.	0.7	-	-	N.D.
64948-4	BP 4-W 5"	-	-	N.D.	0.7	-	-	N.D.
64948-5	BP 5-W 5"	-	-	N.D.	0.7	-	-	N.D.
64948-6	BP 6-W 5"	-	-	N.D.	0.7	-	-	N.D.

* FOR FIBERS > 10um ONLY

The analysis was carried out to the approved TEM method. This laboratory is in compliance with the quality specified by the method.

B m Kell
Authorized Signature

**Analysis of Water by Transmission Electron Microscopy
(EPA-600 R 94 134)**

EMS No. 64948

Client

Johns Manville Tech. Center

Sample No. BP 1-W 5

Date Analyzed

4/18/00

Fibers > 10 µm in length (chrysotile)

BDL*

MFL

Mass (chrysotile)

0

ug/L

More/Less than 5 Fibers
in Sample (chrysotile)

LESS

Poisson 95% Confidence Interval

0 to 2.7

MFL

Detection Limit

0.7

MFL

* BDL : Below Detection Limit; MFL: Million Fibers per Liter

Particle Size Distribution (Chrysotile)

Particle Length - Microns

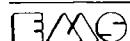
O - 0.49	0.50 - 0.99	1.00 - 1.49	1.50 - 1.99	2.00 - 2.49	2.5 - 4.99	5.00 - 9.99	10 & UP
0	0	0	0	0	0	0	0

Particle Width - Microns

O - .04	.05 - .09	.1 - .14	.15 - .19	.2 - .24	.25 - .49	.50 - .99	1 & UP
0	0	0	0	0	0	0	0

Aspect Ratio L/W

0 - 9.9	10 - 19.9	20 - 29.9	30 - 39.9	40 - 49.9	50 - 99	100 - 199	200 & UP
0	0	0	0	0	0	0	0



RECEIVING

Prüfung

ANALYSIS

[illegible]

Clean <input type="checkbox"/>	Other _____					
Debris <input type="checkbox"/>	Very Light <input type="checkbox"/>	Light <input type="checkbox"/>	Moderate <input checked="" type="checkbox"/>	Heavy <input type="checkbox"/>	Very Heavy <input type="checkbox"/>	
Gypsum <input type="checkbox"/>	Very Light <input type="checkbox"/>	Light <input type="checkbox"/>	Moderate <input type="checkbox"/>	Heavy <input type="checkbox"/>	Very Heavy <input type="checkbox"/>	

RECEIVING

EMS Lab No. 64948
Page _____ of _____

H600A - Serial No. 542-24-03 ☒

H600B - Serial No. 542-05-06 ☐

H600C - Serial No. 542-36-01 ☐

Grid Address B

Screen Magnification 9.4x

Camera Constant 29.8

Accelerating Voltage 100 KV

Beam Current 10 μA

K-Factor 1.5

Analyst Radtke Date 4/18

ANALYSIS

TEM - 1B (11-99)

Clean ☐

Debris: ☐

Gypsum: ☐

Condition of the Grid: ☐

Very Light ☐

Very Light ☐

Good ☐

Light ☐

Light ☐

Scrappy ☐

Moderate ☒

Moderate ☒

Undissolved Filter ☐

Heavy ☐Heavy ☐

Folded ☐

Very Heavy ☐

Very Heavy ☐

RECEIVING

EMS Lab No. 64948
Page _____ of _____

H600C - Serial No. 542-36-01 ☐

Analyst S. Ahmed Date 4/18/00

ANALYSIS

[illegible]

Very Heavy ☐

**Analysis of Water by Transmission Electron Microscopy
(EPA-600 R 94 134)**

EMS No.	64948	Client	Johns Manville Tech. Center
Sample No.	BP 2-W 5	Date Analyzed	4/18/00

Fibers > 10 μ m in length (chrysotile)	<u>BDL*</u>	MFL
Mass (chrysotile)	<u>0</u>	ug/L
More/Less than 5 Fibers in Sample (chrysotile)	<u>LESS</u>	
Poisson 95% Confidence Interval	<u>0 to 2.7</u>	MFL
Detection Limit	<u>0.7</u>	MFL

* BDL : Below Detection Limit; MFL: Million Fibers per Liter

Particle Size Distribution (Chrysotile)

Particle Length - Microns							
O - 0.49	0.50 - 0.99	1.00 - 1.49	1.50 - 1.99	2.00 - 2.49	2.5 - 4.99	5.00 - 9.99	10 & UP
<u>0</u>	<u>0</u>	<u>0</u>	<u>0</u>	<u>0</u>	<u>0</u>	<u>0</u>	<u>0</u>
Particle Width - Microns							
O - .04	.05 - .09	.1 - .14	.15 - .19	.2 - .24	.25 - .49	.50 - .99	1 & UP
<u>0</u>	<u>0</u>	<u>0</u>	<u>0</u>	<u>0</u>	<u>0</u>	<u>0</u>	<u>0</u>
Aspect Ratio L/W							
0 - 9.9	10 - 19.9	20 - 29.9	30 - 39.9	40 - 49.9	50 - 99	100 - 199	200 & UP
<u>0</u>	<u>0</u>	<u>0</u>	<u>0</u>	<u>0</u>	<u>0</u>	<u>0</u>	<u>0</u>

RECEIVING

EMS Lab No. 64948
Page _____ of _____

2024

ANALYSIS

Analyst W. J. L. Date 4-16-60

[illegible]

Very Heavy ☐

RECEIVING

EMS Lab No. 64948
Page _____ of _____

H600C - Serial No. 542-36-01 ☐

Analyst RAJL Date 4/18

ANALYSIS

IS: Clean ☐ Debris: ☐ Very Light ☐ Light ☐ Moderate ☒ Heavy ☐ Very Heavy ☐
Gypsum: ☐ Very Light ☐ Light ☐ Moderate ☒ Heavy ☐ Very Heavy ☐
Condition of the Grid: ☐ Good ☐ Scrappy ☐ Undissolved Filter ☐ Folded ☐

RECEIVING

ANALYSIS

H600C - Serial No. 542-36-01 ☐

Analyst S. Ahmed Date 11/17/00

[illegible]

Very Heavy ☐

**Analysis of Water by Transmission Electron Microscopy
(EPA-600 R 94 134)**

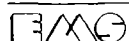
EMS No.	64948	Client	Johns Manville Tech. Center
Sample No.	BP 3-W 5	Date Analyzed	4/18/00

Fibers > 10 μ m in length (chrysotile)	<u>BDL*</u>	MFL
Mass (chrysotile)	<u>0</u>	ug/L
More/Less than 5 Fibers in Sample (chrysotile)	<u>LESS</u>	
Poisson 95% Confidence Interval	<u>0 to 2.7</u>	MFL
Detection Limit	<u>0.7</u>	MFL

* BDL : Below Detection Limit; MFL: Million Fibers per Liter

Particle Size Distribution (Chrysotile)

Particle Length - Microns							
O - 0.49	0.50 - 0.99	1.00 - 1.49	1.50 - 1.99	2.00 - 2.49	2.5 - 4.99	5.00 - 9.99	10 & UP
<u>0</u>	<u>0</u>	<u>0</u>	<u>0</u>	<u>0</u>	<u>0</u>	<u>0</u>	<u>0</u>
Particle Width - Microns							
O - .04	.05 - .09	.1 - .14	.15 - .19	.2 - .24	.25 - .49	.50 - .99	1 & UP
<u>0</u>	<u>0</u>	<u>0</u>	<u>0</u>	<u>0</u>	<u>0</u>	<u>0</u>	<u>0</u>
Aspect Ratio L/W							
0 - 9.9	10 - 19.9	20 - 29.9	30 - 39.9	40 - 49.9	50 - 99	100 - 199	200 & UP
<u>0</u>	<u>0</u>	<u>0</u>	<u>0</u>	<u>0</u>	<u>0</u>	<u>0</u>	<u>0</u>



RECEIVING

EMS Lab No. 64948
Page _____ of _____

PRP

ANALYSIS

Analyst W. J. Date 4-16-00

[illegible]

Clean <input type="checkbox"/>	Other _____				
Debris <input type="checkbox"/>	Very Light <input type="checkbox"/>	Light <input type="checkbox"/>	Moderate <input checked="" type="checkbox"/>	Heavy <input type="checkbox"/>	Very Heavy <input type="checkbox"/>
Gypsum <input type="checkbox"/>	Very Light <input type="checkbox"/>	Light <input type="checkbox"/>	Moderate <input type="checkbox"/>	Heavy <input type="checkbox"/>	Very Heavy <input type="checkbox"/>

RECEIVING

ANALYSIS

H600C - Serial No. 542-36-01 ☐

Analyst Kach Date 9/18

TEM - 1B (11-99)

Very Heavy ☐

RECEIVING

ANALYSIS

H600A - Serial No. 542-24-03 ☐

H600B - Serial No. 542-05-06 ☒

H600C - Serial No. 542-36-01 ☐

K-Factor 1.18
Analyst S. Ahmed Date 4/18/00

[illegible]

IS: Clean ☐ Debris: ☐ Very Light ☐ Light ☐ Moderate ☐ Heavy ☐ Very Heavy ☐
Gypsum: ☐ Very Light ☐ Light ☐ Moderate ☐ Heavy ☐ Very Heavy ☐
Condition of the Grid: ☐ Good ☒ Scrappy ☐ Undissolved Filter ☐ Folded ☐

**Analysis of Water by Transmission Electron Microscopy
(EPA-600 R 94 134)**

EMS No.	64948	Client	Johns Manville Tech. Center
Sample No.	BP 4-W 5	Date Analyzed	4/18/00

Fibers > 10 μ m in length (chrysotile)	<u>BDL*</u>	MFL
Mass (chrysotile)	<u>0</u>	ug/L
More/Less than 5 Fibers in Sample (chrysotile)	<u>LESS</u>	
Poisson 95% Confidence Interval	<u>0 to 2.7</u>	MFL
Detection Limit	<u>0.7</u>	MFL

* BDL : Below Detection Limit; MFL: Million Fibers per Liter

Particle Size Distribution (Chrysotile)

Particle Length - Microns							
O - 0.49	0.50 - 0.99	1.00 - 1.49	1.50 - 1.99	2.00 - 2.49	2.5 - 4.99	5.00 - 9.99	10 & UP
<u>0</u>	<u>0</u>	<u>0</u>	<u>0</u>	<u>0</u>	<u>0</u>	<u>0</u>	<u>0</u>
Particle Width - Microns							
O - .04	.05 - .09	.1 - .14	.15 - .19	.2 - .24	.25 - .49	.50 - .99	1 & UP
<u>0</u>	<u>0</u>	<u>0</u>	<u>0</u>	<u>0</u>	<u>0</u>	<u>0</u>	<u>0</u>
Aspect Ratio L/W							
0 - 9.9	10 - 19.9	20 - 29.9	30 - 39.9	40 - 49.9	50 - 99	100 - 199	200 & UP
<u>0</u>	<u>0</u>	<u>0</u>	<u>0</u>	<u>0</u>	<u>0</u>	<u>0</u>	<u>0</u>

RECEIVING

EMS Lab No. 64948
Page _____ of _____

H600A - Serial No. 542-24-03 ☒

H600B - Serial No. 542-05-06 ☐

H600C - Serial No. 542-36-01 ☐

Grid Address B
Screen Magnification 9770 X
Camera Constant 28.1
Accelerating Voltage 100 KV
Beam Current 10 μ A
K-Factor 1.5
Analyst Redke Date 4/1/8

ANALYSIS

[illegible]**OBSERVATIONS:**

IS: Clean ☐ Debris: ☐ Very Light ☐ Light ☐ Moderate ☒ Heavy ☐ Very Heavy ☐
Gypsum: ☐ Very Light ☐ Light ☐ Moderate ☐ Heavy ☐ Very Heavy ☐
Condition of the Grid: ☐ Good ☐ Scrappy ☐ Undissolved Filter ☐ Folded ☐

RECEIVING

ANALYSIS

H600C - Serial No. 542-36-01 ☐

Analyst S. H. Ahmed Date 2/18/00

[illegible]

S: Clean ☐ Debris: ☒ Very Light ☐ Light ☐ Moderate ☐ Heavy ☒ Very Heavy ☐
Gypsum: ☐ Very Light ☐ Light ☐ Moderate ☐ Heavy ☐ Very Heavy ☐
Condition of the Grid: ☐ Good ☒ Scrappy ☐ Undissolved Filter ☐ Folded ☐

**Analysis of Water by Transmission Electron Microscopy
(EPA-600 R 94 134)**

EMS No. 64948

Client

Johns Manville Tech. Center

Sample No. BP 5-W 5

Date Analyzed

4/18/00

Fibers > 10 µm in length (chrysotile)

BDL*

MFL

Mass (chrysotile)

0

ug/L

More/Less than 5 Fibers
in Sample (chrysotile)

LESS

Poisson 95% Confidence Interval

0 to 2.7

MFL

Detection Limit

0.7

MFL

* BDL : Below Detection Limit; MFL: Million Fibers per Liter

Particle Size Distribution (Chrysotile)

Particle Length - Microns

O - 0.49	0.50 - 0.99	1.00 - 1.49	1.50 - 1.99	2.00 - 2.49	2.5 - 4.99	5.00 - 9.99	10 & UP
0	0	0	0	0	0	0	0

Particle Width - Microns

O - .04	.05 - .09	.1 - .14	.15 - .19	.2 - .24	.25 - .49	.50 - .99	1 & UP
0	0	0	0	0	0	0	0

Aspect Ratio L/W

0 - 9.9	10 - 19.9	20 - 29.9	30 - 39.9	40 - 49.9	50 - 99	100 - 199	200 & UP
0	0	0	0	0	0	0	0

RECEIVING

EMS Lab No. 64948
Page _____ of _____

ASPECT RATIO
3:1 ☒ 5:1 ☐

Approved By: W. J. C.

FILTER TYPE / AREA (mm²)

MCE	<input type="checkbox"/>	385	<input type="checkbox"/>
PC	<input checked="" type="checkbox"/>	314	<input type="checkbox"/>
MCN	<input type="checkbox"/>	1017	<input type="checkbox"/>
Other			

PORE SIZE

0.45 μm ☐ 0.8 μm ☐
0.1 μm ☒ 0.22 μm ☐
Other _____

G.O. Area (mm²) 0.0 0.02
No. of G.O. to Analyze 10
Filter Lot No. _____

INDIRECT PREP ☐
Somerset
 10:30 - 11 AM
 Bedford: 12:05 PM
 4-13-00

Volume _____ liters
Working Volume 15 ml
Weight _____ grams
Ashed Area _____ %

Prepared By W.S.
Date 4-14-00

MICROSCOPE

Serial No. 542-05-06 H600A ☐

Serial No. 542-05-13 H600B ☐

Grid Address 4
Screen Magnification 9900 X
Camera Constant 28.1
Accelerating Voltage 100 KV
Beam Current 10 μ m
K-Factor 1.8

Analyst W. J. Date 4-16-60

PRDP

ANALYSIS

[illegible]**OBSERVATIONS:**

Clean <input type="checkbox"/>	Other _____				
Debris <input type="checkbox"/>	Very Light <input type="checkbox"/>	Light <input type="checkbox"/>	Moderate <input checked="" type="checkbox"/>	Heavy <input type="checkbox"/>	Very Heavy <input type="checkbox"/>
Gypsum <input type="checkbox"/>	Very Light <input type="checkbox"/>	Light <input type="checkbox"/>	Moderate <input type="checkbox"/>	Heavy <input type="checkbox"/>	Very Heavy <input type="checkbox"/>

RECEIVING

ANALYSIS

H600C - Serial No. 542-36-01 ☐

Analyst Rachha Date 4/18

[illegible]

S: Clean ☐
Debris: ☐ Very Light ☐ Light ☐ Moderate ☒ Heavy ☐ Very Heavy ☐
Gypsum: ☐ Very Light ☐ Light ☐ Moderate ☐ Heavy ☐ Very Heavy ☐
Condition of the Grid: ☐ Good ☐ Scrappy ☐ Undissolved Filter ☐ Folded ☐

RECEIVING

EMS Lab No. 64948
Page _____ of _____

H600C - Serial No. 542-36-01 ☐

Analyst S. H. Hing Date 6/18/00

ANALYSIS

Very Heavy ☐

**Analysis of Water by Transmission Electron Microscopy
(EPA-600 R 94 134)**

EMS No.	64948	Client	Johns Manville Tech. Center	
Sample No.	BP 6-W 5	Date Analyzed	4/18/00	

Fibers > 10 μ m in length (chrysotile)	<u>BDL*</u>	MFL
Mass (chrysotile)	<u>0</u>	ug/L
More/Less than 5 Fibers in Sample (chrysotile)	<u>LESS</u>	
Poisson 95% Confidence Interval	<u>0 to 2.7</u>	MFL
Detection Limit	<u>0.7</u>	MFL

* BDL : Below Detection Limit; MFL: Million Fibers per Liter

Particle Size Distribution (Chrysotile)

Particle Length - Microns							
O - 0.49	0.50 - 0.99	1.00 - 1.49	1.50 - 1.99	2.00 - 2.49	2.5 - 4.99	5.00 - 9.99	10 & UP
<u>0</u>	<u>0</u>	<u>0</u>	<u>0</u>	<u>0</u>	<u>0</u>	<u>0</u>	<u>0</u>
Particle Width - Microns							
O - .04	.05 - .09	.1 - .14	.15 - .19	.2 - .24	.25 - .49	.50 - .99	1 & UP
<u>0</u>	<u>0</u>	<u>0</u>	<u>0</u>	<u>0</u>	<u>0</u>	<u>0</u>	<u>0</u>
Aspect Ratio L/W							
0 - 9.9	10 - 19.9	20 - 29.9	30 - 39.9	40 - 49.9	50 - 99	100 - 199	200 & UP
<u>0</u>	<u>0</u>	<u>0</u>	<u>0</u>	<u>0</u>	<u>0</u>	<u>0</u>	<u>0</u>

RECEIVING

EMS Lab No. 64948
Page _____ of _____

MICROSCOPE
Serial No. 542-05-06 H600A ☐
Serial No. 542-05-13 H600B ☒

Grid Address A
Screen Magnification 9100 X
Camera Constant 28.1
Accelerating Voltage 100 KV
Beam Current _____ μ m
K-Factor 1.8

Analyst W.T. Date 4-16-00

Prepared By W. J.
Date 4-14-00

PARCE

ANALYSIS

[illegible]**OBSERVATIONS:**

Clean <input type="checkbox"/>	Other _____				
Debris <input type="checkbox"/>	Very Light <input type="checkbox"/>	Light <input type="checkbox"/>	Moderate <input checked="" type="checkbox"/>	Heavy <input type="checkbox"/>	Very Heavy <input type="checkbox"/>
Gypsum <input type="checkbox"/>	Very Light <input type="checkbox"/>	Light <input type="checkbox"/>	Moderate <input type="checkbox"/>	Heavy <input type="checkbox"/>	Very Heavy <input type="checkbox"/>

RECEIVING

ANALYSIS

Grid Address B
Screen Magnification 740 X
Camera Constant 29.6
Accelerating Voltage 100 KV
Beam Current 10 μ A
K-Factor 1.5
Analyst Rack Date 4/1/80

[illegible]

IS: Clean ☐ Debris: ☐ Very Light ☐ Light ☐ Moderate ☐ Heavy ☒ Very Heavy ☐
Gypsum: ☐ Very Light ☐ Light ☐ Moderate ☐ Heavy ☐ Very Heavy ☐
Condition of the Grid: ☐ Good ☐ Scrappy ☐ Undissolved Filter ☐ Folded ☐

RECEIVING

ANALYSIS

H600C - Serial No. 542-36-01 ☐

K-Factor 1.8

Analyst S Ahmed Date 4/18/00

[illegible]

Very Heavy ☐

**Analysis of Water by Transmission Electron Microscopy
(EPA-600/4-83-043)**

EMS No. 64948 Date Analyzed 4/16/00

Client Johns Manville Tech. Center

Sample No. EMS Blank

Fibers (chrysotile)	<u>ND</u>	MFL
> 5 Micron length (chrysotile)	<u>ND</u>	MFL
Mass (chrysotile)	<u>0</u>	ug/L
More/Less than 5 Fibers in Sample (chrysotile)	<u>LESS</u>	
Sensitivity Level	<u>0.01</u>	MFL

Particle Size Distribution (Chrysotile)

Particle Length - Microns

O -0.49	0.50 - 0.99	1.00 - 1.49	1.50 - 1.99	2.00 - 2.49	2.5 & UP
<u>0</u>	<u>0</u>	<u>0</u>	<u>0</u>	<u>0</u>	<u>0</u>

Particle Width - Microns

O - .04	.05 - .09	.1 - .14	.15 - .19	.2 - .24	.25 & UP
<u>0</u>	<u>0</u>	<u>0</u>	<u>0</u>	<u>0</u>	<u>0</u>

Aspect Ratio L/W

0 - 9.9	10 - 19.9	20 - 29.9	30 - 39.9	40 - 49.9	50 & UP
<u>0</u>	<u>0</u>	<u>0</u>	<u>0</u>	<u>0</u>	<u>0</u>



RECEIVING

EMS Lab No. 07170
Page _____ of _____

MICROSCOPE
Serial No. 542-05-06 H600A ☒
Serial No. 542-05-13 H600B ☒

Grid Address B
Screen Magnification 1940 X
Camera Constant 29.7
Accelerating Voltage 100 KV
Beam Current 10 μ m
K-Factor 1.5
Analyst Rade Date 4/1/8

[illegible]

Clean <input type="checkbox"/>	Other _____				
Debris <input type="checkbox"/>	Very Light <input type="checkbox"/>	Light <input checked="" type="checkbox"/>	Moderate <input type="checkbox"/>	Heavy <input type="checkbox"/>	Very Heavy <input type="checkbox"/>
Gypsum <input type="checkbox"/>	Very Light <input type="checkbox"/>	Light <input type="checkbox"/>	Moderate <input type="checkbox"/>	Heavy <input type="checkbox"/>	Very Heavy <input type="checkbox"/>



Johns Manville

Chain of Custody

Sample Submission Form

Johns Manville Technical Center
Industrial Hygiene Laboratory
10100 West Ute Avenue
Littleton, CO 80127-5002
303-978-3724
303 978-3005 (fax)

JM Project NO: _____

CLIENT INFORMATION	BILLING INFORMATION	SERVICE NEEDS
Company: <u>CADUS, Inc.</u>	Company: <u>Johns Manville Corp.</u>	7 Day (Standard): <u>X</u>
Address: <u>1121 E Main St.</u>	Address: <u>1871 N. Pershing Rd</u>	RUSH (*) _____
<u>Suite 150</u>		-5 Day _____
City State, Zip: <u>St. Charles IL 60174</u>	City State, Zip: <u>Waukegan IL 60087</u>	3 Day _____
Client Contact: <u>Bill Bow</u>	Client Contact: <u>Glen Bowman</u>	48 Hours _____
Phone No: <u>(630) 587-5306</u>	Phone No: <u>(847) 360-4444</u>	24 Hours _____
Fax No: <u>(630) 587-5348</u>	Fax No: <u>(847) 360-4558</u>	(*) Call for Prices and Availability

Client Reference No: _____

Purchase Order No.: _____

Sampled By: John Chittwood / CADUS

Asbestos

NO	SAMPLE NUMBER	SAMPLE DESCRIPTION	SAMPLE DATE	SAMPLING TIME (min)	SAMPLING RATE (L/min)	SAMPLE VOLUME (L)	MEDIA TYPE*
1	BP1-W5	Water	4/12	0945			X
2	BP2-W5	Water	4/12	1015			X
3	BP3-W5	Water	4/12	1035			X
4	BP4-W5	Water	4/12	1050			X
5	BP6-W5	Water	4/12	1105			X
6	BP5-W5	Water	4/12	1120			X
7							
8							
9							
10							
11							
12							
13							
14							
15							

*: Any bulk material remaining after analysis will be returned to the client submitting the material.

COMMENTS: FedEx Airbill 816304255360

EMS Laboratories,

117 West Bellevue Dr.

Pasadena CA 91105

Nicky Ameli (626) 568-4065

RELINQUISHED BY: <u>John Chittwood</u>	TIME/DATE: <u>1600 4/12/00</u>	RELINQUISHED TO: <u>FedEx / Rec. EMS Lab</u>	TIME/DATE: <u>7:27 4/12/00</u>
RELINQUISHED BY: _____	TIME/DATE: _____	RELINQUISHED TO: _____	TIME/DATE: _____
RELINQUISHED BY: _____	TIME/DATE: _____	RELINQUISHED TO: _____	TIME/DATE: _____

Instructions for completion on reverse side.

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JM: